# AQUATIC EFFECTS TECHNOLOGY EVALUATION (AETE) PROGRAM

**Cost-effective protocols for the collection,**
**filtration and preservation of surface waters**
**for detection of metals and metalloids**
**at ppb (µg $l^{-1}$) and ppt (ng $l^{-1}$) levels**

**Phase I: Evaluation of Bottle Type, Bottle**
**Cleaning, Filter and Preservation Technique**

**AETE Project 3.1.3**

# Cost-effective protocols for the collection, filtration and preservation of surface waters for detection of metals and metalloids at ppb ($\mu g\ l^{-1}$) and ppt ($ng\ l^{-1}$) levels

## Phase I: Evaluation of Bottle Type, Bottle Cleaning, Filter and Preservation Technique

*Submitted to*

Aquatic Effects Technology Evaluation Program

*By*

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# AQUATIC EFFECTS TECHNOLOGY EVALUATION PROGRAM

## Notice to Readers

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### Cost-effective protocols for the collection, filtration and preservation of surface waters for detection of metals and metalloids at ppb ($\mu g\ l^{-1}$) and ppt ($ng\ l^{-1}$) levels

### Phase I: Evaluation of Bottle Type, Bottle Cleaning, Filter and Preservation Technique

The Aquatic Effects Technology Evaluation (AETE) program was established to review appropriate technologies for assessing the impacts of mine effluents on the aquatic environment. AETE is a cooperative program between the Canadian mining industry, several federal government departments and a number of provincial governments; it is coordinated by the Canada Centre for Mineral and Energy Technology (CANMET). The program is designed to be of direct benefit to the industry, and to government. Through technical and field evaluations, it will identify cost-effective technologies to meet environmental monitoring requirements. The program includes three main areas: acute and sublethal toxicity testing, biological monitoring in receiving waters, and water and sediment monitoring.

The technical evaluations are conducted to document certain tools selected by AETE members, and to provide the rationale for doing a field evaluation of the tools or provide specific guidance on field application of a method. In some cases, the technical evaluations include a go/no go recommendation that AETE takes into consideration before a field evaluation of a given method is conducted.

The technical evaluations are published although they do not necessarily reflect the views of the participants in the AETE Program. The technical evaluations should be considered as working documents rather than comprehensive literature reviews.

The purpose of the technical evaluations is to document specific monitoring tools. AETE committee members would like to stress that no one single tool can provide all the information required for a full understanding of environmental effects in the aquatic environment.

For more information on the monitoring techniques, the results from their field application and the final recommendations from the program, please consult the AETE Synthesis Report to be published in February 1999.

Any comments concerning the content of this report should be directed to:

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101-02577/

# PROGRAMME D'ÉVALUATION DES TECHNIQUES DE MESURE D'IMPACTS EN MILIEU AQUATIQUE

## Avis aux lecteurs

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### Protocoles efficaces par rapport au coût pour la collecte, la filtration et la conservation des eaux de surface aux fins de la détection de métaux et de métalloïdes à des niveau ppb ($\mu g\ l^{-1}$) et ppt ($ng\ l^{-1}$)

### Phase I : évaluation du type de bouteille, du nettoyage des bouteilles et du filtre, et de la méthode de conservation

Le Programme d'évaluation des techniques de mesure d'impacts en milieu aquatique (ÉTIMA) vise à évaluer les différentes méthodes de surveillance des effets des effluents miniers sur les écosystèmes aquatiques. Il est le fruit d'une collaboration entre l'industrie minière du Canada, plusieurs ministères fédéraux et un certain nombre de ministères provinciaux. Sa coordination relève du Centre canadien de la technologie des minéraux et de l'énergie (CANMET). Le programme est conçu pour bénéficier directement aux entreprises minières ainsi qu'aux gouvernements. Par des évaluations techniques et des études de terrain, il permettra d'évaluer et de déterminer, dans une perspective coût-efficacité, les techniques qui permettent de respecter les exigences en matière de surveillance de l'environnement. Le programme comporte les trois grands volets suivants : évaluation de la toxicité aiguë et sublétale, surveillance des effets biologiques des effluents miniers en eaux réceptrices, et surveillance de la qualité de l'eau et des sédiments.

Les évaluations techniques sont menées dans le but de documenter certains outils de surveillance sélectionnés par les membres d'ÉTIMA et de fournir une justification pour l'évaluation sur le terrain de ces outils ou de fournir des lignes directrices quant à leur application sur le terrain. Dans certains cas, les évaluations techniques pourraient inclure des recommandations relatives à la pertinence d'effectuer une évaluation de terrain que les membres d'ÉTIMA prennent en considération.

Les évaluations techniques sont publiées bien qu'elles ne reflètent pas nécessairement toujours l'opinion des membres d'ÉTIMA. Les évaluations techniques devraient être considérées comme des documents de travail plutôt que des revues de littérature complètes.

Les évaluations techniques visent à documenter des outils particuliers de surveillance. Toutefois, les membres d'ÉTIMA tiennent à souligner que tout outil devrait être utilisé conjointement avec d'autres pour permettre d'obtenir l'information requise pour la compréhension intégrale des impacts environnementaux en milieu aquatique.

Pour des renseignements sur l'ensemble des outils de surveillance, les résultats de leur application sur le terrain et les recommandations finales du programme, veuillez consulter le Rapport de synthèse

ÉTIMA qui sera publié en février 1999.

Les personnes intéressées à faire des commentaires concernant le contenu de ce rapport sont invitées à communiquer avec M$^{me}$ Geneviève Béchard à l'adresse suivante:

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# Executive Summary

## Background

Guidelines and criteria levels for metals and metalloids in surface waters (CCME, BC, Ontario, EPA) are the subject of much discussion currently and there is a movement to (a) lower many of them from ppb to ppt levels and (b) define element species (rather than total) with respect to level. Their measurement at ppt levels is made possible by the establishment of ICP-MS (inductively coupled plasma mass spectrometry) as a routine analytical technique. However, the various sampling and preservation protocols in effect for measurement at the higher ppb levels have not been verified (in the literature) at the lower ppt concentration levels. Some protocols (e.g. Environmental Protection Agency (EPA) Method 1631 for Hg) call for the exclusive use of expensive Teflon bottles for sample collection and storage; these protocols were published when alternative plasticware development was in its infancy. The 'dissolved' fraction of metals is operationally defined as that which passes a 0.45 µm filter. Often filter types (tortuous path, sieve-like), filter systems, and cleaning thereof, are not specified (e.g. in EPA Methods 200.7, 200.8, CLP Method 6020), though the analytical procedure is extremely detailed.

The objective of the work described herein was to recommend the most cost-effective and efficient procedure by which to sample, filter and preserve surface water for the accurate determination of Al, Ag, As, Cd, Co, Cr, Cu, Hg, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl and Zn to 'consensus' water quality guidelines. The work is focussed on the *filtered* (0.45 µm) water sample, not on *'total recoverable'*. Furthermore, it does not enter into speciation of the 'dissolved' fraction but rather the analytical methods used here are designed to measure the 'total dissolved' fraction (with the exception of Se where only inorganic species are measured).

All elements except Hg and Se were measured by conventional nebulisation ICP-MS which provided detection limits (DL) of: 0.03 ppb for Al; 1.7 ppb for Fe; 20 ppt for Cu and Ni; and ≤10 ppt for the other 11 elements. These DLs are well under the consensus criteria levels for waters (information taken from Environment Canada, BC and Ontario). Hydride or vapour generation ICP-MS was used for Hg ($Hg^{0}$) and Se ($SeH_2$) to achieve the required sensitivity of measurement, down to 1 and 4 ppt, respectively. All experimental work prior to analysis was carried out in a Class 100 Cleanroom.

## Results

### *Test-tubes*

The test-tubes recommended for use in the analysis of waters at low analyte concentrations are the Fisherbrand polypropylene centrifuge tubes. These should be soaked in 1% $HNO_3$ for 24 hours and rinsed with water. Their blue polystyrene caps are to be avoided when analysing for Al and Zn, unless these undergo vigorous cleaning. These test-tubes do not require cleaning for the determination of

Hg and Se at levels of 1 ppt or greater.

### *Bottles*

The bottles studied comprise: Teflon (*FEP*, Nalge #1600); high density polyethylene (*HDPE*, Nalge #2007); polyethylene terephthalate copolyester (*PETG*, Nalge #2019); polypropylene (*PP*, Nalge #2006); and precleaned HDPE$^P$ ('Superfund-Analyzed' to meet or exceed EPA specifications). Two cleaning methods were investigated: a modified EPA Method 1638; and one promoted by the State of Virginia. These procedures are similar in that they focus on the use of $HNO_3$ but the EPA method employs concentrated (12M) acid whereas the Virginian method uses a much lower concentration of 5% (v/v) with a subsequent step employing only 0.5% $HNO_3$. The EPA method incorporates an initial wash with a soap solution. A third method was tested, that of simply rinsing each bottle with deionised water three times prior to filling. Each group of five bottles, cleaned in three different ways, was filled to 125 ml with 0.4% $HNO_3$ (usual concentration of acid as preservative employed by the Geological Survey of Canada (GSC) and others) and a charge of 1 ml of 8M $HNO_3$ was added to each of the alternate group of five. After six days, they were analysed for all elements save Hg which required a completely separate test as different preservatives are needed for this element.

*The approach practised by some laboratories - to add a charge of concentrated (• 8M)* $HNO_3$ *reagent to the bottle hours or days before water collection - is not acceptable.* This strategy causes much higher levels of contamination for all bottles than is the case when acidifying during or after sample collection. Cleaning does not remove 'available' elements as prolonged contact with $HNO_3$ leaches out significant quantities.

*The least expensive bottle (ca $Cdn 0.90), made of high density polyethylene (HDPE), shows the best characteristics and is highly recommended.* It could be used without rigorous cleaning (only rinsing with deionised water) if batches are checked but a rinse with weak $HNO_3$ (5%) is probably advisable.

HDPE bottles purchased precleaned are an unnecessary expense (ca $Cdn 2.30) as they are *inferior* to their uncleaned counterparts and show a startling increase in Zn contamination, to 739±195 ppt (cf 7±4 ppt). Higher levels of Al, Cr, Ni and Pb are also evident.

Comparable in cost to the HDPE is the *polypropylene bottle (PP) which does require cleaning* if Al is of concern (contamination level of 594±40 ppt). Cleaning by either method reduces this consistent level of contamination to insignificance. Considerably more expensive at $Cdn 2.60, the polyethylene terephthalate copolyester (PETG) bottle must also be cleaned, but by the 5% $HNO_3$ method.

*The extremely expensive ($28) Teflon (FEP) is not recommended.* It was, by far, the dirtiest bottle. Both methods of cleaning adequately reduce contamination by Cr, Fe, Co, Ni, Cu, Zn, Mo and Pb for environmental projects but concern remains for some of these elements (e.g. Cr and Ni) if geochemical mapping is the focus.

Overall, results indicate that the less costly method of cleaning, as outlined by the State of Virginia, is preferable to the EPA method. This method could probably be shortened by eliminating the second 24-hour stage of contact with 0.5% $HNO_3$.

All bottle types - FEP, HDPE, PETG and PP - can be used without any cleaning (i.e. rinsing only) for the determination of Hg in waters down to levels of 1-2 ppt. None of the commonly used preservation media for Hg - 0.5% BrCl, 2% HCl or 0.04% $K_2Cr_2O_7$ in 0.1% $HNO_3$ - appears to leach out detectable concentrations of these elements from the bottle material. *Thus, the elaborate cleaning methods, EPA 1631 and 1638, can be avoided for Hg.*

### *Filter systems*

The majority of the twelve 0.45 µm and two 5 µm filters tested (of syringe, in-line and vacuum type) were from two leading manufacturers, Gelman and Millipore. The objective of this project was two-fold in that the expected contamination levels for both an ordinary water sample (e.g. stream) and an acidic sample (e.g. end-of-pipe) were desired. The test media were Type I deionised water and 0.4% $HNO_3$.

*Overall, optimum performance in terms of contamination and ease of use was achieved with the ion chromatography Acrodisc syringe filter with Supor membrane, from Gelman and the Sterivex syringe filter capsule with Durapore membrane, from Millipore*. Nylon membranes should be avoided as they are slow and do not show superior contaminant characteristics. The Millex LS 5µm syringe prefilter is recommended for samples high in particulate matter. Also acceptable for environmental monitoring are: the Millicup bottle top with Durapore membrane (vacuum system); the in-line Gelman AquaPrep with Thermopor membrane; and the AquaPrep 250 with Supor membrane. For the filtration of acidic samples, the following systems should be avoided: the Gelman syringe nylon Acrodisc; the Millipore all-glass vacuum; the Gelman groundwater capsule; and the Gelman AquaPrep 250.

These filters were investigated further - for their propensity to retain elements which are present as colloids which should pass through a 0.45 µm pore size. A bulk control sample of Ottawa River water and a synthetic spiked water sample were employed for this purpose. Although the Acrodisc Supor membrane from Gelman was recommended for its low contamination level, recoveries of certain analytes in Ottawa River water are significantly lower than those obtained using the Millipore systems which incorporate the Durapore membrane. This indicates partial retention of the elements Al, Cr, Fe, Mn, Co, Zn and Pb present in colloidal form. The high and consistent recoveries for all 17 elements found in the Ottawa River control with the Millipore systems advocate *recommendation of the Sterivex capsule system or Durapore-based alternatives if the goal is to measure that fraction of an element present at • 0.45 µm.* Lowest recoveries, and hence maximum retention of colloidal species, were found with Gelman's Supor membrane-based systems.

*Further assessment of the sorption of Hg, in its free ion form, by different filter systems is required.* Unlike the other 16 elements, spikes of Hg added to deionised water samples were not fully recovered through all filter systems. Minimum loss is evident using the Millipore systems, particularly the Millicup bottle top model with Durapore membrane.

### *Stability*

Four samples of different matrix - Ottawa, Rideau and Gatineau River waters and a spiked water sample - were used to verify that preservation of the 16 analytes (i.e. all except Hg) in a medium of 0.4% $HNO_3$ was adequate for prolonged storage (one month).

*Acidification to 0.4% in $HNO_3$ should maintain elements Al, As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl and Zn in water samples for at least a month at room temperature. Stability over this period for Ag at concentrations of several hundred ppt is questionable and matrix-dependent. Stability of these elements is independent of container material.*

*The best preservation reagent for Hg is 0.5% BrCl*: it maintains Hg at ppt levels in solution for at least one month. Preservation in 2% HCl or 0.04% $K_2Cr_2O_7$ may be inadequate, especially for the former medium. Stability of Hg was independent of the container material. Diffusion of Hg from the atmosphere into the sample was not in evidence over the 28-day trial period for any of the bottle types.

# Sommaire

## Contexte

Les lignes directrices et les normes applicables aux concentrations de métaux et de métalloïdes dans les eaux de surface (CCME, C.-B., Ontario, EPA) sont actuellement remises en question, la majorité des parties intéressées convenant de la nécessité de réduire bon nombre des concentrations limites de x parties par milliard à x parties par billion et de définir des valeurs limites par espèce d'élément plutôt que des valeurs limites totales. Le recours systématique à la spectrométrie d'émission de plasma induit par haute fréquence (SE/PIHF) permet de repousser les limites de la détection jusqu'à des concentrations de l'ordre de quelques parties par billion. Toutefois, l'efficacité des différents protocoles d'échantillonnage et de conservation qui régissent actuellement la mesure de concentrations de métaux et de métalloïdes comprises dans la fourchette supérieure des parties par milliard n'a pas été vérifiée (dans la littérature) à des concentrations plus faibles comprises dans la fourchette inférieure des parties par billion. Certains protocoles (p. ex. méthode 1631 de l'EPA pour le Hg) prévoient l'usage exclusif de flacons en téflon fort dispendieux pour le prélèvement et l'entreposage des échantillons d'eau. Il convient toutefois de noter qu'au moment de la publication de ces protocoles, la gamme d'articles en plastique disponibles était encore extrêmement limitée. La fraction « dissoute » des métaux est définie en pratique comme étant la fraction qui traverse un filtre à mailles de 0,45 µm. Souvent, ces protocoles ne contiennent aucune spécification concernant le type de filtre (à trajectoire tortueuse, tamis) ou les systèmes de filtration et d'épuration (p. ex. méthodes 200.7 et 200.8 de l'EPA, méthode 6020 du programme de contact de laboratoire) à utiliser, alors que la méthode d'analyse proposée est décrite dans les moindres détails.

L'objectif des travaux décrits dans les pages qui suivent consistait à recommander la méthode la plus rentable (dans une perspective coût-efficacité) et la plus efficiente pour prélever, filtrer et conserver des échantillons d'eau de surface en vue de mesurer avec précision les concentrations de Al, Ag, As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Mo, Ni, Pb, Sb, Se, Tl et Zn et, en bout de ligne, uniformiser les lignes directrices relatives à la qualité de l'eau. Les recommandations formulées ci-après s'appliquent aux échantillons d'eau *filtrés* (0,45 µm), et non pas au « total récupérable ». Enfin, les méthodes analytiques décrites ici ne visent pas à établir la composition de la fraction dissoute, mais plutôt à mesurer la fraction totale dissoute (à l'exception du Se, dont seules les formes inorganiques sont mesurées).

Le dosage de tous les éléments à l'exception du Hg et du Se a été effectué par SE/PIHF classique par nébulisation. Cette méthode permet de repousser les limites de détection à 0,03 parties par milliard pour l'Al, à 1,7 parties par milliard pour le Fe, à 20 parties par billion pour le Cu et le Ni et à ≤10 parties par billion pour les 11 autres éléments. Ces limites sont nettement inférieures aux limites fixées pour l'eau (source des informations : EnvCan, C.-B. et Ontario). Pour le dosage du Hg ($Hg^0$) et du Se ($SeH_2$), on a dû recourir à une version hybride de SE/PIHF par génération de vapeur pour obtenir des mesures suffisamment sensibles, les valeurs limites s'établissant à respectivement 1 et 4 parties par billion. Toutes les étapes expérimentales préliminaires aux analyses se sont déroulées dans une salle propre de classe 100.

## Résultats

### *Éprouvettes*

Les éprouvettes recommandées pour l'analyse des échantillons d'eau renfermant de faibles concentrations de contaminants sont les tubes à centrifugation en polypropylène distribués par la société Fisherbrand. Ces éprouvettes doivent subir un trempage dans du $HNO_3$ à 1% pendant 24 heures puis un rinçage dans l'eau avant d'être utilisées. L'utilisation des bouchons en polystyrène bleu durant le dosage de l'Al et du Zn est contre-indiquée, sous réserve d'un nettoyage minutieux au préalable. Aucun nettoyage des éprouvettes n'est nécessaire pour le dosage du Hg et du Se à des concentrations égales ou supérieures à 1 partie par billion.

### *Bouteilles*

Les cinq types de bouteilles suivants ont été évalués : téflon (*FEP*, Nalge #1600); polyéthylène haute densité (*HDPE*, Nalge #2007); copolyester de polyéthylène téréphthalate (*PETG*, Nalge #2019); polypropylène (*PP*, Nalge #2006); $HDPE^P$ avec prénettoyage (analysé en vertu de la *Loi Superfund* pour respecter ou dépasser les spécifications de l'EPA). Deux méthodes de nettoyage ont été évaluées : une variante de la méthode 1638 de l'EPA; et une méthode proposée par l'État de Virginie. Ces deux méthodes se ressemblent dans la mesure où elles prévoient toutes deux l'utilisation de $HNO_3$; la méthode de l'EPA préconise toutefois l'utilisation d'acide concentré (12M), tandis que la méthode de la Virginie utilise dans un premier temps une concentration beaucoup plus faible de 5% (v/v) et, dans un deuxième temps, du $HNO_3$ à seulement 0,5%. La méthode de l'EPA comporte en outre un lavage initial avec une solution savonneuse. Une troisième méthode, consistant simplement à rincer trois fois chaque bouteille avec de l'eau désionisée avant le remplissage, a également été évaluée. Des groupes de cinq bouteilles ont été nettoyées selon l'une ou l'autre des trois méthodes susmentionnées. Ensuite, 125 mL de $HNO_3$ à 0,4% (concentration d'acide couramment utilisée à des fins de conservation par la CGC et d'autres établissements) et une charge de 1 mL de $HNO_3$ 8M ont été versés dans chaque groupe de cinq bouteilles. Le dosage de tous les éléments, à l'exception du Hg, a été réalisé six jours plus tard. Le dosage du Hg a été effectué à l'aide d'une méthode complètement différente nécessitant l'emploi d'autres agents de conservation.

*L'approche utilisée par certains laboratoires, qui prévoit l'ajout d'une solution de réactif* $HNO_3$ *concentrée (• 8M) dans la bouteille quelques heures ou quelques jours précédant le prélèvement de l'échantillon d'eau, est jugée inacceptable*. La contamination est beaucoup moins importante si l'acidification survient pendant ou après le prélèvement de l'échantillon. Le nettoyage n'enlève pas les éléments disponibles, alors qu'un contact prolongé avec le $HNO_3$ provoque la lixiviation de quantités importantes d'éléments.

*La bouteille la moins dispendieuse (environ 0,90 $Can), faite de polyéthylène haute densité (HDPE) est également celle qui présente les meilleures caractéristiques. Son utilisation est de ce fait fortement recommandée.* Aucun nettoyage minutieux préalable n'est nécessaire (un simple rinçage à l'eau désionisée suffit) si les lots ont été vérifiés, mais un rinçage avec une solution faible de $HNO_3$ (5%) apparaît indiquée.

L'achat de bouteilles HDPE prénettoyées constitue une dépense inutile (environ 2,30 $Can), car les caractéristiques de ce type de bouteille sont inférieures à celles de la bouteille HDPE non prénettoyée. En outre, le niveau de contamination par le Zn augmente considérablement pour atteindre 739±195 parties par billion (comparativement à 7±4 parties par billion). La même tendance s'observe avec l'Al, le Cr, le Ni et le Pb.

D'un coût comparable à celui de la bouteille HDPE, la *bouteille en polypropylène (PP) nécessite un nettoyage* lorsque l'éventualité d'une contamination par l'Al constitue un problème (niveau de contamination : 594±40 parties par billion). Les deux méthodes de nettoyage permettent de réduire le niveau de contamination à un seuil négligeable. Nettement plus dispendieuse à l'achat (2,60 $Can), la bouteille en copolyester de polyéthylène téréphthalate (PETG) nécessite également un nettoyage, mais selon la méthode prévoyant l'utilisation de $HNO_3$ à 5%.

*La bouteille en téflon n'est pas recommandée en raison de son coût exorbitant.* Parmi les cinq types de bouteilles examinés, c'est de loin la bouteille la moins propre. Les deux méthodes de nettoyage permettent de réduire efficacement le niveau de contamination par le Cr, le Fe, le Co, le Ni, le Cu, le Zn, le Mo et le Pb pour les projets environnementaux. Toutefois, certains éléments (p. ex. Cr et Ni) continuent de poser un problème s'il s'agit d'un projet de cartographie géochimique.

Dans l'ensemble, l'évaluation révèle que la méthode de nettoyage la moins dispendieuse, soit celle proposée par la Virginie, est supérieure à la méthode de l'EPA. L'élimination de la deuxième étape de trempage au $HNO_3$ à 5% permettrait probablement d'accélérer cette méthode.

Tous les types de bouteilles - FEP, HDPE, PETG et PP - peuvent être utilisées sans nettoyage préalable (c'est-à-dire, après un simple rinçage) pour le dosage du Hg dans l'eau à des concentrations aussi faibles que 1 à 2 parties par billion. Aucun des milieux de conservation couramment utilisés pour le Hg - BrCl à 0,5%, HCl à 2% ou $K_2Cr_2O_7$ à 0,04% dans $HNO_3$ à 0,1% - ne semble entraîner un lessivage de concentrations détectables de ces éléments à partir du matériel utilisé pour la fabrication des bouteilles. *Les méthodes de nettoyage 1631 et 1638 de l'EPA, fort complexes, peuvent donc être omises pour le Hg.*

## Systèmes de filtration

La majorité des filtres de 12 à 0,45 µm et de 2 à 5 µm évalués (type seringue, en ligne ou sous vide) provenaient de deux importants fabricants, Gelman et Millipore. L'objectif visé par les évaluateurs était double, dans la mesure où les niveaux de contamination prévus pour l'échantillon d'eau ordinaire (p. ex. cours d'eau) et l'échantillon d'effluent acide (p. ex. point de rejet)] étaient souhaités dans les deux cas. De l'eau désionisée de type I et du $HNO_3$ à 0,4% ont été utilisés comme milieux d'essai.

*De façon générale, les meilleurs rendements en ce qui a trait à la contamination et à la facilité* *d'utilisation ont été observés avec le filtre Acrodisc de type seringue à membrane Supor pour chromatographie d'échange d'ions (Gelman) et la capsule filtrante Sterivex pour seringue à membrane Durapore (Millipore).* Les membranes en nylon sont à éviter, car elles sont lentes et présentent un rendement inférieur en ce qui a trait à la contamination. Le préfiltre de type seringue Millex LS 5 µm est recommandé pour les échantillons renfermant de fortes concentrations de particules. Sont également considérés comme acceptables pour la surveillance environnementale les dispositifs suivants : flacon Millicup à membrane Durapore (système sous vide); système en ligne AquaPrep à membrane Thermopor de Gelman; système AquaPrep 250 à membrane Supor. Pour la filtration des échantillons acides, les systèmes suivants sont à éviter : filtre Acrodisc nylon de type seringue; système sous vide en verre Millipore; capsule pour eaux souterraines Gelman; système AquaPrep 250 de Gelman.

Ces filtres ont également été évalués en fonction de leur capacité de retenir des éléments qui, à l'état colloïdal, devraient pouvoir traverser des pores de 0,45 µm. Un échantillon témoin global prélevé dans l'Outaouais et un échantillon d'eau traité artificiellement ont été utilisés à cette fin. Bien que recommandé en raison de son faible niveau de contamination, le système Acrodisc à membrane Supor de Gelman a présenté un rendement nettement inférieur à celui des systèmes Millipore à membrane Durapore pour ce qui est de la récupération de certains contaminants à partir de l'échantillon d'eau de l'Outaouais. Ces résultats indiquent une rétention partielle des éléments Al, Cr, Fe, Mn, Co, Pb et Zn à l'état colloïdal. En considération des taux de récupération systématiquement élevés obtenus avec les systèmes Millipore pour les 17 éléments présents dans l'échantillon d'eau témoin prélevé dans l'Outaouais, le *système à capsule Sterivex ou les unités filtrantes à membrane Durapore sont recommandés pour la mesure de la fraction d'un élément de taille égale ou inférieure à 0,45 µm.* Les taux de récupération les plus faibles ou, en d'autres mots, les plus forts taux de rétention des formes colloïdales ont été enregistrés avec les systèmes à membrane Supor de Gelman.

*La capacité de sorption du Hg sous sa forme ionique libre des différents systèmes de filtration demeure à évaluer.* Contrairement aux 16 autres éléments, les quantités de Hg ajoutées aux échantillons d'eau désionisée n'ont pas été récupérées entièrement par tous les systèmes de filtration. Des pertes minimales ont été enregistrées avec les systèmes Millipore, en particulier le flacon Millicup munie à membrane Durapore.

### ***Stabilité***

Quatre échantillons d'origine différente - rivières Outaouais, Rideau et Gatineau et eau traitée - ont été utilisés pour vérifier si l'utilisation d'un milieu à base de $HNO_3$ à 0,4% permet de conserver efficacement les 16 éléments (c'est-à-dire, tous les éléments sauf le Hg) durant une période prolongée (un mois).

*L'acidification en présence de* $HNO_3$ *à 0,4% devrait assurer la conservation des éléments Al, As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl et Zn dans les échantillons pendant une période d'au moins un mois à la température de la pièce. Au-delà de cette période, la stabilité de l'Ag à des concentrations de plusieurs centaines de parties par billion est incertaine et dépend de la matrice.*

*Le matériel utilisé pour la fabrication des contenants n'influe pas sur la stabilité de ces éléments.*

*Le meilleur réactif pour la conservation du Hg est le BrCl à 0,5%.* Ce réactif permet de conserver des concentrations de Hg de l'ordre des parties par billion durant au moins un mois. La capacité de conservation du HCl à 2% et, tout particulièrement, du $K_2Cr_2O_7$ pourrait ne pas être satisfaisante.

# 1. Evaluation of test-tubes to be used in this study

Prior to testing bottles for their level of contamination, the test-tubes to be used in various experiments were evaluated for their potential contribution of Ag, Al, As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Mo, Ni, Pb, Sb, Se, Tl and Zn. The 15 ml Fisherbrand disposable, calibrated, centrifuge polypropylene tubes selected (Fisher Scientific P/N 05-539-5) are those commonly employed in inorganic laboratories in Canada. They are made by Corning and are similar to the more expensive Falcon tubes, with blue polystyrene screw caps. A stock solution of 0.4% $HNO_3$ (representative of the acid concentration used by the GSC and others to preserve these elements in water samples) was prepared from ultrapure $HNO_3$ (Seastar Chemicals Inc., double sub-boiling distilled, CA-01-02-1000). Type I water (<18 MΩ) was obtained from a Milli-Q system. All work in this project was carried out in a Class 100 Cleanroom on Class 100 benches or in a Class 10 fume-hood. One hundred test-tubes (as received, not rinsed) were filled with about 10 ml of 0.4% $HNO_3$ and half were inverted after capping so that the dilute acid would be in contact with the cap material. Both sets were allowed to stand for 24 hours and subsequently analysed by pneumatic nebulisation ICP-MS (for method, see Hall *et al.*, 1996a).

A second test was performed on cleaned test-tubes. A suite of 125 tubes was filled with 14 ml of 1% $HNO_3$ (again, Seastar), covered with parafilm and allowed to stand for 48 hours. These tubes were then rinsed with Type I water three times and 10 ml of 0.4% $HNO_3$ were added to each tube for subsequent analysis after a 24-hour period of standing.

The mean and standard deviation for these three sets of data are presented in Table 1.1; these data have not been truncated at instrumental detection limits in order to observe trends. The slightly negative values for most elements determined in the third set of samples (1% $HNO_3$ wash of test-tubes) were caused by low-level contamination of the calibration solutions as they sat in the carousel in the lab. Construction was going on in the corridor and undoubtedly elevated the contamination level of the air. For the most part, the uncleaned test-tubes contributed little in contamination to the 0.4% $HNO_3$ blank level. However, where levels did increase, they did so in several elements. For example, two test-tubes showed elevated levels of Cu (27 and 22 ppt), Zn (271 and 241 ppt) and Pb (18 and 13 ppt). Contact of solution with the cap clearly causes contamination for Al (5.8 ± 1.1 ppb) and Zn (1.0 ± 0.4 ppb) at levels which are too high (maxima of 8.3 and 3.6 ppb, respectively, for Al and Zn) to be acceptable. The contamination level of Mn was raised slightly, from 1 ± 1 to 5 ± 5 ppt, but note that the average instrumental $3\sigma$ detection limit is 5 ppt. Washing the test-tubes with 1% $HNO_3$ had the effect of making the blank levels of Al and Zn much more consistent (with RSDs of 6 and 13%, respectively, compared to 93 and 150% when unwashed). These 125 test-tubes were then rinsed with Type I water, sealed in polyethylene bags and stored in the Cleanroom to be used subsequently for the bottle study.

Determination of Hg and Se required the added sensitivity of hydride generation (HG) ICP-MS, using the method and instrumentation described by Hall and Pelchat (1997a, b). As these elements are determined from a strongly acidic (4-6 M HCl) solution in order to create a reducing environment, this medium was substituted for the 0.4% $HNO_3$ investigated above for the majority of elements studied. Two solutions were tested for Hg: 4 M HCl and 4 M HCl containing 0.5% of the oxidant, BrCl (see later discussion). Fifty test-tubes were filled with 4 M HCl, and another 50 with the added BrCl. After 48 hours, these solutions were analysed. Of the 50 containing 4 M HCl, only one showed a detectable Hg concentration of 2 ppt. None of the 50 containing 4 M HCl and 0.5% BrCl showed Hg levels greater than or equal to 1 ppt. These test-tubes were employed for the subsequent work.

Fifty test-tubes were filled with 6 M HCl and analysed for Se after 48 hours of contact. A 10 ml portion was taken and heated in a water bath for 30 minutes to reduce any Se VI to IV, the solution diluted to 4 M HCl by addition of 5 ml of water, and finally analysed by HG-ICP-MS. None of these 50 samples contained Se at detectable levels (i.e. all at levels <4 ppt).

### *Recommendations*

- For the ultra-trace determination of Ag, Al, As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Tl and Zn in water samples, soak Fisherbrand polypropylene tubes in 1% $HNO_3$ for 24 hours and rinse with water. Caps are to be avoided when analysing for Al and Zn, unless these undergo vigorous cleaning (which has not been investigated here).

- These test-tubes do not require cleaning for the determination of Hg and Se at levels of 1 ppt or greater. However, a brief rinse with dilute HCl (of the same stock as the medium used for analysis) is probably a good precautionary measure.

# 2. Evaluation of bottles for contamination of trace metals/metalloids

A schematic diagram (Figure 2.1) provides a visual summary of the treatment of five types of bottles and the cleaning procedures tested. A review (though not exhaustive) of common practices used by analytical laboratories in Canada led to the choices made in bottle types and cleaning methods investigated. With the exception of the analysis itself, all this work was carried out in a Class 100 Cleanroom.

## 2.1. Bottles investigated

The bottles selected were those in widespread use for the collection and analysis of water samples for metals/metalloids. These comprise the following bottles of nominal 125 ml capacity:

| | |
|---|---|
| FEP | (Teflon) Nalge #1600, round with Tefzel ETFE screw, \$28 each (approx cost Cdn) |
| HDPE | (High density polyethylene) Nalge #2007, rectangular with polypropylene screw closure, \$0.90 each |
| PETG | (Polyethyleneterephthalate copolyester), Nalge #2019, square with HDPE screw closure, \$2.60 each |
| PP | (Polypropylene) Nalge #2006, round with PP screw closure, \$1.00 each |
| HDPE$^{P}$ | Purchased precleaned, round, "Superfund-Analyzed" to meet or exceed EPA specifications, \$2.30 each |

## 2.2. Cleaning methods used for all elements except Hg

The term 'water' throughout refers to Type I (<18MΩ), produced by a Milli-Q system. Ten bottles of each type were cleaned using the three methods outlined below, making a total of 120 bottles (excluding the precleaned HDPE$^{P}$ bottles).

***Cleaning methods:***

***(1) Simple rinse*** Bottle and cap were simply rinsed three times with water before filling or charging with acid.

***(2) Modified EPA*** This basic procedure is outlined in EPA Method 1638, *"Determination of trace metals in ambient waters by ICP-MS"* (EPA 821-R-96-005). However, since the $Cl^-$ ion is undesirable in the measurement of some elements by ICP-MS, the step of soaking in 1 M HCl was eliminated. Method 1638 (p. 25) does allow for modification to the complete cleaning procedure if results prove satisfactory. Briefly, the modified method involves:

- Bottle is filled with a 0.5% solution of liquid detergent (Alconox) and shaken for 30 min. Bottle and cap are then rinsed with water until there is no sign of soap residue.

-Bottle is filled with reagent-grade 16M $HNO_3$ and placed in a water bath at 50°C for 2 h. This was *not* carried out for the PETG bottles as they are not resistant to this concentration of acid and melt in the bath. Therefore, the PETG bottles were filled with 5% (v/v) $HNO_3$ instead and heated.

-Bottle is rinsed thoroughly with water and filled with water. Each bottle is double-bagged in polyethylene and stored in a Class 100 Cleanroom.

***(3) $HNO_3$ wash*** As described in *"Quality Assurance Project Plan for Clean Metals"* (Dec. 1996) by the Office of Water Quality Assessment and Planning, Virginia Dept. of Environmental Quality.

-Bottle is filled with 5% (v/v) $HNO_3$, capped and placed in a water bath for 24 h at 50°C.

-Bottle is well rinsed with water, refilled with 0.5% (v/v) $HNO_3$, capped and placed in a water bath for 24 h at 50°C.

-Bottle is rinsed three times with water, and refilled with water to which 250 µl of Seastar (double-distilled, CA-01-02-1000; Sidney, BC) conc. $HNO_3$ are added. Bottle is capped and set aside for 24 h.

-Bottle is rinsed with water and filled with water until usage (again double-bagged and stored in a Class 100 Cleanroom).

Thus, the two cleaning procedures are similar in that they focus on the use of $HNO_3$ but the EPA method employs concentrated (12M) acid whereas the Virginian method (designated as $HNO_3$ hereafter) uses a much lower concentration of 5% (v/v) with a subsequent step employing only 0.5% $HNO_3$. The EPA method incorporates an initial wash with a soap solution.

## 2.3. Simulated preservation treatment for all elements except Hg

The suite of ten bottles cleaned by each method was divided in two. Each group of five bottles was filled to 125 ml with 0.4% $HNO_3$ (Seastar Chemicals Inc., double sub-boiling distilled, CA-01-02-1000). To the other five bottles, a charge of 1 ml of 8M $HNO_3$ (Seastar) was added. Care was taken to use the same lot of Seastar $HNO_3$ for both treatments. The suite of 10 HDPE$^P$ bottles was treated in the same way (i.e. five were filled to 125 ml with 0.4% $HNO_3$ and five were charged with 1 ml of 8M $HNO_3$). These two approaches were used to imitate procedures used in industry: some companies pre-charge bottles with a small volume of concentrated acid, to which the sample is added later in the field, whereas others prefer to add the acid once the sample is collected. The concentration of 0.4% is used in GSC hydrogeochemical surveys and is a compromise between 0.1 and 1% $HNO_3$, the range of preservative concentration indicated by a scan of reports and literature.

The bottles were allowed to stand for six days, whereupon the 65 bottles containing the 1 ml of 8M acid charges were filled with 124 ml of water. An aliquot was then taken from each bottle and poured into test-tubes ready for analysis. These test-tubes had been already investigated for their contamination levels (see Section 1). The method of direct ICP-MS used to determine Ag, Al, As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Tl and Zn is described in Hall *et al.* (1996a). Calibration was carried out with standard solutions of 0.4% $HNO_3$ (same Seastar source as used previously) spiked with known amounts of element standard solutions. Selenium required improved sensitivity by changing the method of introduction, from pneumatic nebulisation to hydride generation. This method is described in Hall and Pelchat (1997a, b).

## 2.4. Results for all elements except Hg and Se

The mean and standard deviation of element concentrations found in the 125 ml volume of water standing in each bottle are presented in Table 2.1. The instrumental $3\sigma$ detection limits (DL) shown are based on 'pure' solutions and consequently are the most optimistic: these would be degraded when analysing 'real' waters where oxide corrections etc would be made for certain elements (e.g. CaO, CaOH on Ni and Co). Values have not been truncated at the DL in order to view trends. All the data are attached in Appendix 2.1 and mean results are summarised in histogram form in Figure 2.2. Some general observations can be gleaned from a quick scan of this figure:

! acid-charged bottles contribute much more contamination than their counterparts containing 0.4% $HNO_3$;

! FEP bottles are the most contaminating;

! the modified EPA method of cleaning is inferior to that proposed by the State of Virginia; and

! the precleaned $HDPE^P$ bottles are inferior to their less expensive, untreated HDPE counterparts.

In greater detail, with reference to Table 2.1 and Figure 2.2:

***Al*** The "dirtiest" bottle by far is PP, with a maximum level of contamination of 4.2 ± 0.2 **ppb** being shown by the uncleaned, acid-charged bottle. Neither the EPA nor the $HNO_3$ method of cleaning is adequate to completely remove this contamination for the acid-charged bottle (B), though they are acceptable for the dilute acidified sample (A). The cleanest bottle is PETG, showing levels of Al all below the instrumental DL of 30 ppt. Both cleaning methods are satisfactory for FEP, though the level of contamination 'as is' (rinse only) is minimal (52 ppt for bottle A and 176 ppt for B). The $HDPE^P$ bottle shows the same level of Al contamination, at 130-150 ppt, for bottles A and B, whereas the 'ordinary' HDPE bottle shows measurable levels of contamination only when in prolonged contact with the acid charge and then at a consistent 281±19 ppt. Al in HDPE bottle A is below the DL of 30 ppt.

***Cr*** The HDPE, PETG and PP bottles all show negligible levels of Cr at several ppt, though the acid-charged bottle does display slightly higher levels, as does EPA cleaning (to ca 20 ppt). Compared to the HDPE bottle, the HDPE$^{P}$ bottle A shows a higher level of Cr, at 35 ± 6 ppt, which increases to 102 ± 23 ppt for bottle B. Maximum contamination is observed for the FEP bottle B, at 218 ± 117 ppt, with the A counterpart indicating lower levels of 67 ± 32 ppt. These concentrations are reduced substantially by HNO$_3$ cleaning but the EPA method is slightly inferior, showing mean values of 44 and 74 ppt, respectively for bottles A and B.

***Fe*** Contamination by Fe is severe in the FEP bottle, at 1.9 ± 1.1 ppb, rising to 42 ± 60 **ppb** in the acid-charged bottle. This contamination in bottle B is random, the range being from several ppb to 140 ppb. Both cleaning methods decrease this level below 1 ppb in bottle A, though the acid-charged bottle still shows 1-2 ppb Fe (equivalent to the DL of the method). The other bottles show less than 1 ppb of Fe contamination with the exception of the acid-charged PETG bottle at 1.2 ± 0.9 ppb. Precleaned HDPE$^{P}$ is not cleaner than HDPE in Fe.

***Mn*** The PETG bottle shows the highest degree of Mn contamination, at 67 ± 7 ppt in A and rising to 5.3 ± 1.1 **ppb** in the acid-charged bottle B. Both cleaning methods eliminate this contamination from bottle A but are not successful in doing so with bottle B, with the level of Mn remaining at the several ppb level. The other bottle of concern is the FEP, showing 162 ± 201 ppt Mn in the acid-charged bottle; however, cleaning by either method reduces this below 10 ppt. All HDPE and PP bottles show Mn levels below 10 ppt.

***Co*** Again, PETG bottles, followed by FEP, contribute most contamination. A high amount of Co is extracted from the acid-charged PETG bottle (9.1 ± 1.9 **ppb,** cf to 121 ± 12 ppb in bottle A). This potential contamination in bottle B is **not** removed by either the EPA nor HNO$_3$ cleaning methods, though bottle A Co levels are reduced to several ppt. The difference in results between bottles A and B is less startling for the FEP material, at 30 and 69 ppt Co, respectively. However the cleaning methods do not completely eliminate these contributions, especially in the acid-charged bottles. All HDPE and PP bottles show <3 ppt Co.

***Ni*** FEP bottles show an unacceptable and random level of Ni contamination which is of the same magnitude in bottle A and B, at ca 500 ppt. Cleaning with HNO$_3$ eliminates this contribution of Ni (to <20 ppt) for treatment A but not B (remaining at a mean of 233 ppt). The EPA cleaning method is not adequate for either the dilute acidified sample A (126 ppt) or the acid-charged bottle B (245 ppt). Nickel contamination in other bottles is negligible.

***Cu*** FEP bottles again contribute high and varied levels of Cu (A-190 ± 199 ppt; B-1.6 ± 2.0 **ppb**) in the unwashed bottles. Both methods of cleaning reduce these levels to below 50 ppt. Similarly, both methods negate the levels of Cu observed in the HDPE, PETG and PP bottles but these levels are much lower, in the range 20-90 ppt Cu. Precleaned HDPE$^{P}$ bottles show about the same level of Cu as the HDPE (14-32 ppt).

***Zn*** The dirtiest bottles are the precleaned HDPE$^P$, contributing 739 ± 195 ppt Zn to bottle A and 2.0 ± 1.2 **ppb** to bottle B. The cleanest bottle is the HDPE, at <20 ppt Zn, followed by the PP bottles at <30 ppt Zn. Though the cleaning methods reduce the contribution of Zn from FEP and PETG bottles down from averages of 98-766 ppt, random contamination is still evident (e.g. to 750 ppt in one of the FEP bottles, A).

***As*** No contamination above 10 ppt is evident for As in these bottles.

***Mo*** Only the FEP bottles show a contribution by Mo above the DL of 3 ppt, at 9 ± 4 ppt in bottle A and 164 ± 254 ppt in the acid-charged bottle, B. Both cleaning methods reduce the level below 4 ppt in the dilute acid treatment, A, but slight traces of Mo remain in the acid-charged bottles (16-20 ppt).

***Ag*** No contamination above the DL of 2 ppt is evident for Ag in these bottles.

***Cd*** Only the acid-charged, uncleaned FEP bottle shows a mean value for Cd above the DL of 2 ppt and this is at a level of only 3 ± 1 ppt. The EPA method reduces this below the DL (to 1 ± 0.7 ppt, and <1 ppt for the dilute acid equivalent).

***Sb*** Levels of Sb are below the DL of 3 ppt in precleaned and ordinary HDPE and PP bottles. PETG shows the highest contamination, at 3 ppt on average for bottle A and 14 ppt for bottle B. Neither cleaning method reduces this level adequately for the acid-charged bottle.

***Tl*** All Tl concentrations are below 1 ppt.

***Pb*** Mean levels of Pb are below the DL of 2 ppt in HDPE, PETG and PP bottles. However, the HDPE$^P$ shows 4 ± 2 ppt Pb in both treatments. The FEP bottles are again the dirtiest, at 10 ± 11 ppt in bottle A and 64 ± 59 ppt in bottle B. These levels are reduced to several ppt by cleaning.

## 2.5. Testing of bottles for Se contamination

The selenium test was carried out on all the bottles prepared as described in Sections 2.2 and 2.3 but hydride generation (HG) ICP-MS was used to ensure adequate sensitivity of measurement. The HG system is outlined in Figure 2.3; measurement was made at isotopes 77 and 78. Only Se IV is reactive to form the gaseous hydride and therefore any Se VI was prereduced to the IV valency state by heating in 6M HCl. A 5 ml aliquot of solution was added to 5 ml of 12 M HCl in a test-tube, capped, shaken and heated in a water bath for 35 minutes. On cooling, the solution was made up to 15 ml with water and analysed by HG-ICP-MS. Results for the 130 bottles are attached in Appendix 2.2: all mean values for Se are below the method detection limit of 4 ppt.

## 2.6. Testing of bottles for Hg contamination

A completely different set of bottles, though of the same types, was used for Hg testing as the preservation techniques required for this element differ from the 0.4% $HNO_3$ medium used for most cations. The cleaning methods used for these bottles also differed from those described earlier. A schematic representative of this investigation is presented in Figure 2.4. Fifteen bottles of each type were subjected to each of the cleaning methods outlined below.

### 2.6.1. Cleaning methods - Hg

HCl is used both for cleaning bottles and in the determination of Hg, and thus its contamination level for Hg is important. Three different grades of HCl had been tested in previous work. The lowest grade, Baker-analysed reagent-grade, was found to be equivalent in Hg content to the more expensive Ultrex and trace metal purified grades. A concentration of 4 M was found to contain about 3 ppt Hg. The cleaning methods used for Hg comprise:

***(1) EPA - Method 1631***

This procedure is based on that described in EPA Method 1631: *"Mercury in water by oxidation, purge and trap, and cold vapour atomic fluorescence spectrometry"* (EPA 821-R-96-001). Briefly, this involves:
-Bottle is filled with Baker-analysed (reagent-grade) 4 M HCl and placed in a water bath at 70°C for 48 h.
-Bottle is cooled and rinsed three times with water.
-Bottle is filled with 1% (v/v) HCl, capped and placed in an oven at 65°C overnight.
-Bottle is cooled, rinsed three times with 0.4% (v/v) HCl and finally water and dried on a Class 100 bench. It is capped and double-bagged (new polyethylene, zip-locked) until needed.
*Note: PETG bottles deform in the oven.*

***(2) EPA - Method 1638***

This is outlined in EPA Method 1638, *"Determination of trace metals in ambient waters by ICP-MS"* (EPA 821-R-96-005). Briefly, this involves:
- Bottle is filled with 0.5% solution of liquid detergent (Alconox) and shaken for 30 min. Bottle and cap are then rinsed with water until there is no sign of soap residue.
-Bottle is filled with reagent-grade 12M $HNO_3$ and placed in a water bath at 50°C for 2 h. This was *not* carried out for the PETG bottles as

they are not resistant to this concentration of acid and melt in the bath. Therefore, the PETG bottles were filled with 5% $HNO_3$ instead and heated.

-Bottle is rinsed thoroughly with water, filled with 1 M HCl and placed in a water bath at 50°C for 48 h.

-Bottle is rinsed thoroughly with water and filled with 0.1% (v/v) HCl. Each bottle was double-bagged and stored in a Class 100 Cleanroom until used.

***(3) Simple rinse*** Bottle and cap are rinsed three times with water. Bottles are air-dried on a Class 100 bench and used immediately. The precleaned HDPE$^P$ bottles were added to this suite.

### 2.6.2. Preservation methods - Hg

As outlined in Figure 2.4, in conjunction with the bottle testing, three preservation methods were investigated:

***0.5% BrCl*** This is the reagent recommended in Method 1631 to be used for preservation of total dissolved Hg when speciation of the methyl form is not desired. The stock reagent is made as follows:

-27 g of reagent-grade KBr are dissolved in 2.5 l of HCl (cleanest and most economic grade for Hg which has been found to be Baker-analysed) with the aid of stirring (magnetic bar) for ca 1 h in a fume-hood.

-38 g of reagent-grade $KBrO_3$ are added slowly while stirring. This forms a strong oxidising mixture of $Br_2$, $Cl_2$ and BrCl which is then capped, ready for use.

An aliquot of 1.25 ml of the 1:1 reagent was added to each bottle and left for six days after which 124 ml of water were added and the resulting solution analysed.

***2% HCl*** EPA Method 1631 recommends preservation of Hg in a medium of 0.5% HCl when species-specific analysis is required. Other concentrations of HCl have been reported (e.g. Copeland *et al.*, 1996); we chose to investigate Hg stability in 2% HCl. Thus, a charge of 5 ml of 6M HCl was added to each bottle. After six days, 120 ml of water were added and the solution analysed.

***0.04% $K_2Cr_2O_7$*** This is the reagent recommended in the *Standard Methods for the Examination of Water and Wastewater*, APHA-AWWA-WEF (18th edition, Washington, 1992). A solution containing 20% (m/V) of $K_2Cr_2O_7$ in 1:1 $HNO_3$ was prepared and 250 µl pipetted into each 125 ml bottle. After six days, 125 ml of water were added and the solution analysed.

Prior to analysis, an aliquot of 5 ml of each solution was placed in a test-tube containing 2.5 ml of 12

M HCl (to make a solution 4 M in HCl). The BrCl-preserved samples were treated slightly differently in that an aliquot of 20 μl of 30% $NH_2OH.HCl$ (reducing agent to counteract BrCl) was also added. The same configuration as that shown in Figure 2.3 was used; measurement was made at m/z 198 and 202, and calibration was performed using solutions of Hg in 4M HCl.

### 2.6.3. Results - Hg

The results, taken as raw data, are summarised in Table 2.2 and attached in Appendix 2.3. The instrumental detection limit for Hg, based on 3σ of the signal for 20 blank 4 M HCl solutions, is 1 ppt. Again, the data have not been truncated at this DL to observe trends. For all four bottle types, there is no significant difference in results for solutions preserved in 0.5% BrCl or 2% HCl: there is no detectable Hg above 1 ppt in cleaned or uncleaned bottles. Solutions preserved in 0.04% $K_2Cr_2O_7$ show a consistent background Hg contribution of about 1.5 ppt Hg. The slightly lower Hg values (0.6 ppt) in cleaned PP bottles perhaps suggest that some degree of readsorption is occurring in these bottles.

## 2.7. Discussion and Conclusion

The approach practised by some laboratories - to add a charge of concentrated (≥8M) $HNO_3$ reagent to the bottle hours or days before water collection - *is not acceptable*. This strategy causes much higher levels of contamination (for all bottles) than is the case when acidifying during or after sample collection. This occurs in the uncleaned bottles for the elements Al (in FEP, PP, HDPE), Cr (FEP and precleaned $HDPE^P$), Fe (FEP, PETG and precleaned $HDPE^P$), Mn (PETG), Co (FEP and PETG), Ni (PETG), Cu (FEP), Zn (FEP and precleaned $HDPE^P$), Mo (FEP) and Pb (FEP). While the two cleaning methods tested serve to reduce these levels of contamination in the acid-charged bottles, they are not eliminated and elements are leached out during prolonged contact (six-day period used here) with the strong acid. Significant levels remain after cleaning for elements such as Fe and Ni in FEP, Mn and Co in PETG, and Al in PP and HDPE bottles. Thus, a 2-h soaking in 16M $HNO_3$ at 50°C (EPA method) has not effectively removed these elements from these materials, and subsequent extended contact with 1 ml of 8M $HNO_3$ causes more element to be released. Time of contact between acid charge and bottle will clearly vary in a sampling program and thus contaminant level is not predictable.

In order to evaluate whether the contamination levels summarised in Table 2.1 are significant, we must know the minimum concentration of element to be expected in a natural water sample. Data from two stream surveys in Nova Scotia and Newfoundland carried out by the GSC (Hall, 1993; Hall *et al.*, 1994) have been used to provide values of the mean and standard deviation of these elements in their 'dissolved' (defined as <0.45 μm) form. Most of these 868 sites are second- and third-order streams remote from industry or farming, sampled to assess the control of underlying geology on the water chemistry. These values, together with the lowest concentration measured in each survey, are presented in Table 2.3. These data have been used to estimate the method detection limit required

('MDL - geo') in hydrogeochemical surveys, thereby ensuring that the lowest element concentration expected in a survey could be determined with reasonable precision. Also given in Table 2.3 are the detection limits required by typical environmental regulations. For the most part, as expected, the MDLs for environmental assessment purposes are significantly higher than those required to measure natural, geogenic levels of trace elements. However, there are several instances where these two limits are equivalent and this warrants examination. For example, the environmental MDL for Al, at 0.5 ppb, appears to be much too low when compared to the natural range of Al in surface waters. Only 2 samples out of 729 in the Nova Scotia survey contained Al below the analytical detection limit of 2 ppb. The value for the 25th percentile of this survey is 47 ppb and that for the median is 114 ppb. Thus, the requirement to be able to measure at 0.5 ppb for Al in environmental assessment projects seems overly stringent. Similarly, only 2 of the 729 samples contained Cu below the analytical detection limit of 0.1 ppb, the lowest level of measurement required in environmental work. However, the value for the 95th percentile is only 1.5 ppb Cu, demonstrating a much narrower range in natural levels than Al. The mean for Cu in the Newfoundland survey is considerably higher (2.7 ppb, cf 0.7 ppb) due to some base metal mineralisation in the area. Although both environmental and geochemical MDLs for Cr and Cd are comparable, the natural levels of these elements are close to these limits and therefore the seemingly low environmental MDL may not be excessively rigorous.

The MDLs in Table 2.3 have been used to evaluate whether potential levels of contamination from the different bottles could be significant, in either a geochemical or environmental application. For each bottle type and cleaning treatment, the value for the 'mean plus two standard deviations' (taken from Table 2.1) has been compared with the MDL listed in Table 2.3. This exercise pertains only to the bottle filled with 0.4% $HNO_3$, not the acid-charged series. Table 2.4 indicates those elements where this value exceeds the MDL, and therefore where positive significant errors could be expected. Elements are not included where their mean, 'raw' values in Table 2.1 are below the analytical detection limit.

It is clear from this table that:

- The FEP bottles are by far the dirtiest and cleaning, for either application, is mandatory. Both methods of cleaning adequately reduce contamination by Cr, Fe, Co, Ni, Cu, Zn, Mo and Pb for environmental projects but concern remains for some of these elements (e.g. Cr and Ni) if geochemical mapping is the focus.
- Only the HDPE bottle performs well using this criterion of mean plus two standard deviations. The only element possibly of concern in uncleaned HDPE bottles is Ag, but this is due to one bottle which measured 3 ppt (analytical 3σ detection limit is 2 ppt), the others registering less than 0.2 ppt. Cleaning does not demonstrate a real improvement and hence is probably unnecessary for HDPE bottles. In fact, Al levels are raised slightly in these bottles after cleaning by the EPA method.
- The 'precleaned' $\text{HDPE}^{P}$ bottles are definitely not worth the added cost (at least double) as they are, indeed, *inferior* to their uncleaned counterparts and show a startling increase in Zn contamination, to 739±195 ppt (cf 7±4 ppt). Higher levels of Al, Cr, Ni and Pb are also evident.
- The unwashed PETG bottles show significant levels of Co, Cu, Zn and Sb but the bottles cleaned using 5% $HNO_3$ (note PETG does not withstand concentrated $HNO_3$ and heat) show acceptably

low levels of all elements.

! The only element of concern for environmental studies in the unwashed PP bottles is Al, at a contamination level of 594 ± 40 ppt. Cleaning by either method reduces this consistent level of contamination to insignificance. For hydrogeochemical purposes, contamination by Cu, Ag and Sb in the unwashed bottles is almost acceptable and is easily removed by cleaning.

All bottle types - FEP, HDPE, PETG and PP - can be used without any cleaning (i.e. rinsing only) for the determination of Hg and Se in waters down to levels of 1-2 ppt. None of the commonly used preserving agents for these elements ($HNO_3$ for Se; BrCl, HCl or $K_2Cr_2O_7$ in $HNO_3$ for Hg) appears to leach out detectable concentrations of these elements from the bottle material. Thus, the elaborate cleaning methods, EPA 1631 and 1638, can be avoided for Hg.

Some regulatory agencies specify the use of Teflon or glass bottles for collection of samples for subsequent Hg determination because these materials are purported to be superior in their transmission characteristics of volatile $Hg^0$. For example, EPA Method 1631 calls for a combination of fluoropolymer bottles with fluoropolymer-lined caps to preclude contamination from diffusion of atmospheric Hg into the bottle. However, work described later in this report shows that any changes (actually minimal) in Hg concentration in the test water samples over a 28-day period were independent of bottle material. If there was significant concentration of Hg in the atmosphere and it was penetrating through the HDPE, PETG or PP bottle, one would have expected to see an increase in the measured Hg concentration in the preserved water sample; this was not the case. Bottles of standard Hg solutions are often not of glass or Teflon. For example, Delta (Charleston, South Carolina, USA) provides their Hg standard solutions in HDPE bottles, and Spex (Metuchen, New Jersey, USA) supplies their Claritas PPT standards in LDPE. There is a growing amount of evidence in the literature demonstrating that this concern for Hg vapour transmission under ambient conditions is unfounded. For example, Copeland *et al.* (1996) found no difference between PETG and glass bottles for the storage of potable water samples for Hg; the study was carried out over a 10-day period. No significant differences were found by Meranger *et al.* (1981) in their study of Hg in blood after 28 days of storage in soda glass, pyrex glass or polyethylene containers. Daniels and Wigfield (1991) studied 14 different types of tubing for gas-phase adsorption losses of elemental Hg. They found insignificant loss for PTFE, quartz, PP and nylon but 20-29% loss for Tygon, polyvinyl chloride (PVC), LDPE and polyurethane, and 100% loss for silastic tubing. It has been the experience at the GSC that polyurethane and Teflon tubing, used in the determination by HG-ICP-MS, show similar resistance to Hg vapour, and are superior to Tygon. Clearly, this is a somewhat controversial subject where further work, with well defined objectives, is needed.

## *Recommendations*

For the elements studied in this project, the least expensive bottle (ca $Cdn 0.90), made of high density polyethylene (HDPE), shows the best characteristics and could be used without rigorous cleaning (only rinsing in triplicate with deionised water) if batches are checked. HDPE bottles purchased precleaned are an unnecessary expense. Comparable in cost is the polypropylene (PP) bottle which does require cleaning if Al is of concern. Considerably more expensive at $Cdn 2.60, the polyethylene terephthalate copolyester (PETG) bottle must also be cleaned, but by the 5% $HNO_3$ method. The extremely expensive ($28) Teflon (FEP) is **not** recommended. Overall, results indicate that the less costly method of cleaning, as outlined by the State of Virginia, is preferable to the EPA method. This method could probably be shortened by eliminating the second 24-hour stage of contact with 0.5% $HNO_3$.

# 3. Evaluation of filter systems for contamination

Prior to testing a suite of filter systems, the syringes used with some of these systems had to be investigated for their contribution to contamination of the elements under study.

## 3.1. Contamination by syringes used for filtering

Two types of syringes were evaluated: the Becton Dickinson (BD) 60 ml unit with Luer lock (P/N 309663) and rubber-tipped plunger; and the all-plastic Whatman 50 ml unit (P/N 6603-2016 from Delta Scientific). The former syringe is used widely in medical applications whereas the latter is designed mainly for environmental projects.

One set of syringes, comprising three of each type, was prerinsed three times with Type I water (our usual protocol applied to laboratory ware) and then filled with 50 ml of water. This sample was then taken and analysed following acidification to 0.4% $HNO_3$ (Seastar double-distilled, as in Section 2). Another set (n=3) of each type was treated the same way except that 0.4% $HNO_3$ was substituted for water as the test medium. Contact with a moderately weak solution of $HNO_3$ was tested in order to estimate the potential degree of contamination when acidic samples are to be handled, as would be the case in collecting acid mine drainage or end-of-pipe samples. These samples were split into three sets of test-tubes, to be analysed by: (1) nebulisation ICP-MS for the 15-element suite; (2) HG-ICP-MS for Se; and (3) vapour generation-ICP-MS for Hg. The samples for Hg were stabilised by addition of BrCl to a concentration of 0.5% BrCl. As before, the sample for Se determination was treated with HCl (to 6 M HCl) and heated to reduce any Se VI to Se IV prior to analysis by HG-ICP-MS.

Selenium and Hg were not detected (i.e. <4 and 1 ppt, respectively) in any of the 12 test samples.

The values for the mean and standard deviation of the 15-element suite in these test samples are given in Table 3.1. Again, the significance of these potential contributions to a real sample can be assessed by comparing the value for the mean plus two standard deviations with the required method detection limits for geochemical and environmental studies shown in Table 2.3. For the water samples collected following a triplicate rinsing of the syringe, both the BD and Whatman plastic syringes are acceptable for environmental work, focussing on all 15 elements. However, there would be a slight concern in a geochemical survey type of application for low levels of contamination of Zn using the BD syringe (0.4 ± 0.4 ppb, cf MDL of 0.1 ppb). As expected, the acidified water sample caused much higher levels of some elements to be leached out of the syringes but the only concern in an environmental study would be for Al, Cr, Cu and Zn using the BD model. While contributions of Cr and Cu are relatively low and that for Al is acceptable in view of natural ranges (Table 2.3), the high and random contamination of Zn (119 ± 36 ppb) rules out the use of Becton Dickinson syringes for filtration of acidic water samples.

The Whatman plastic syringes were used for the subsequent filtration study.

## 3.2. Evaluation of filter systems

The majority of filters tested were from two leading manufacturers: Gelman and Millipore. Three types of systems were used: syringe (S), in-line (L) and vacuum (V). In total, twelve 0.45 μm and two 5μm filters were evaluated for their potential contamination levels; their designations and brief descriptions are given in Table 3.2. The objective of this project was two-fold in that the expected contamination levels for both an ordinary water sample (e.g. stream) and an acidic sample (e.g. end-of-pipe) were desired. Hence the test media were, again, Type I water and 0.4% $HNO_3$. The procedures used, carried out in triplicate for each filter, are as follows:

***Syringe filters (S)*** These filters were all 25 mm in diameter. A 50 ml aliquot of Type I water was flushed through the filter and collected in a rinsed HDPE bottle for analysis. A further 50 ml aliquot was flushed through and discarded. Finally, a 50 ml aliquot of 0.4% $HNO_3$ was passed through and collected in a separate bottle.

***In-line filters (L)*** These filters, of greater capacity than those above, were flushed initially with 100 ml of water which was retained for analysis. An aliquot of 900 ml was then rinsed through and discarded. A further 100 ml aliquot was passed through and retained for analysis. The filter was then emptied of water by forcing air through the filter (with the syringe). Finally, 100 ml of 0.4% $HNO_3$ were flushed through and retained.

***Vacuum filters (V)*** These filters were all 47 mm in diameter. Each vacuum apparatus was thoroughly rinsed with water. An aliquot of 100 ml of water was then filtered and retained for analysis. An aliquot of 300 ml was filtered and discarded. A further 100 ml aliquot was passed through and retained for analysis. Finally, 100 ml of 0.4% $HNO_3$ were flushed through and retained in a third HDPE bottle. The 5μm M-3-V filter was tested only once rather than three times as it was the same material as its 0.45 μm M-2-V counterpart.

These test samples were split into three portions, as previously, for the separate determination of the 15 element-suite, Se and Hg. The water samples were preserved immediately, with $HNO_3$ (to 0.4%) for the 15-element suite and Se, and with BrCl (to 0.5%) for Hg. As before, the sample for Se determination was treated with HCl and heated to reduce any Se VI to Se IV prior to analysis by HG-ICP-MS.

## 3.3. Results of filter contamination study

The complete dataset, for all 17 elements, is attached as Appendix 3.1. All of the data for Hg and Se in the water and acid rinses were at or, more commonly, below the analytical method detection limits of 1 and 4 ppt, respectively. The values for the mean and standard deviation (n=3) for the suite of 15 elements in the water and acidic water rinses of the 14 filter systems are presented in Table 3.3.

Again, the significance of these potential contributions to a real sample can be assessed by comparing the value for the mean plus two standard deviations with the required method detection limits (lowest concentrations of interest) for geochemical and environmental studies shown in Table 2.3. Cases which are significant are underlined in Table 3.3 and summarised in Table 3.4 where elements of potential concern in geochemical or environmental applications are shown.

With respect to water samples collected for environmental projects, there are seven 0.45 µm filter systems which are acceptable: G-1-S, G-2-S, M-1-S, M-4-S, M-1-V, G-2-L and G-3-L. Of the remaining filters, G-3-S, M-2-S and F-1-V are close to being acceptable as the contaminants (Cd, Cu, Pb) were just at significant levels in the first wash of 50 ml (for S-type) or 100 ml (for V-type). This leaves only filters M-2-V and G-1-L as rejected for their contributions of Al and Cu. The levels of these contaminants are reduced with washing (see data for W2 aliquots) to insignificant values for G-1-L but not for the Millipore system, M-2-V. The vacuum system employed for the M-2-V and M-3-V work was new and it was thought that washing with $HNO_3$ and subsequent processing of the 0.4% $HNO_3$ test solution caused leaching of these elements from the glass surface. These levels of Al and Cu have not been observed in previous work with well-used apparatus. Further support for confidence in this system and the HAWP-type filter paper is the fact that the data for the F-1-V system are much lower (Tables 3.3, 3.4): the same filter paper was used there.

The stringent criteria by which the filter systems are evaluated for use in geochemical surveys results in rejection of most of them (Table 3.4). However, with consideration to the expected natural ranges of these elements (Table 2.3), the following systems would be acceptable: G-1-S, G-3-S, M-1-S, M-2-S, M-4-S, and G-3-L. However, of these, G-3-L should be avoided where samples are likely to be acidic. The poor performance of the G-1-L capsule filter from Gelman, compared to that of the Acrodisc G-1-S, is probably caused by the filter housing material as both incorporate the same Supor membrane. From a practical aspect, the G-2-S Gelman nylon syringe filter is extremely slow, even with these clean water samples, a surprising fact given that it has been designed for use with high particulate or viscous samples. Though not as slow as the Gelman version, the Millipore nylon syringe filter (M-4-S) is inferior to the others in speed. The AquaPrep in-line filter made of Thermopor (G-2-L) should be avoided if Sb is an important element; after 1000 ml of rinsing, there is still a relatively high concentration remaining (34±7 ppt for W2, cf 292±19 ppt for W1). The Sterivex syringe filters (M-1-S) were easiest to use throughout. The capsule filters (e.g. G-1-L) have a large void volume which requires a greater volume of sample as an initial rinse. As a prefilter, the 5µm M-3-S performs well.

## *Recommendations*

! Overall, optimum performance was achieved with the ion chromatography Acrodisc syringe filter from Gelman (G-1-S) and the Sterivex syringe filter capsule from Millipore (M-1-S). Nylon membranes should be avoided as they are slow and do not show superior contaminant characteristics. The Millex LS 5µm syringe prefilter (M-3-S) is recommended for samples high in particulate matter.

- Also acceptable for environmental monitoring are: the Millicup bottle top with Durapore membrane (vacuum system); the in-line Gelman AquaPrep with Thermopor membrane; and the AquaPrep 250 with Supor membrane. With sufficient rinsing, these systems could also be employed: the Gelman syringe GHP Acrodisc; the Millipore Millex HV syringe; and the in-line Gelman groundwater capsule.

- For the filtration of acidic samples, the following systems should be avoided: the Gelman syringe nylon Acrodisc; the Millipore all-glass vacuum; the Gelman groundwater capsule; and the Gelman AquaPrep 250.

These recommendations are based upon the contamination contributed by these filters and their ease of use. However, another parameter - their propensity for clogging and retaining elements - must first be evaluated before final endorsement is given.

## 4. Evaluation of filter systems for retention of colloids

Previous collaborative projects between the GSC and USGS (United States Geological Survey) have indicated that simple filtration of unspecified volumes of natural waters through unspecified 0.45 µm membrane filters may not represent an acceptable operationally-defined definition for 'dissolved' constituents. Hall *et al.* (1996b) reported the study of the effect of four different 0.45 µm pore size filter membrane systems on the 'dissolved' concentration of 28 elements in five natural water samples of varying matrix. In three of the five waters (e.g. St. Lawrence), consistently higher concentrations of most elements (minor and trace) were obtained using the Nucleopore 47 mm filter and the cellulose acetate/nitrate 47 mm filter than those measured using the 142 mm cellulose nitrate MFS filter or the Gelman capsule 47 mm filter. These distinct and coherent patterns in elemental behaviour disappeared for the other two samples, an organic-rich peat water of high suspended load and a mineralised sample high in Si and Ca. Thus, the nature and degree of filtration artifact are matrix-dependent. Horowitz *et al.* (1996) found that the principal cause of these variable recoveries is due to the inclusion/exclusion of colloidally-associated trace elements in the filtrate, although dilution and sorption/desorption from filters may also be factors. Filtration of the Mississippi and Tangipahoa River waters through four different systems resulted in different values for the 'dissolved' concentration of Al, Fe, Mn, Co, Ni, Cu and Zn; Sr, Ba, Ca, Mg, Na and Si showed consistent results. This distinction in element behaviour is not surprising as colloidal species, in the size range 10-500 nm, are known to be comprised of clay, $Fe_2O_3$, $MnO_2$ and humic acid particles to which elements such as Cu and Zn may be adsorbed.

Ottawa River water was used to further evaluate the filter systems under study here for the retention of colloids.

### 4.1. Method

An 8-l sample of Ottawa River water was collected in a HDPE Carboy. A 100-ml aliquot of this unfiltered sample was transferred to a 125-ml HDPE bottle and acidified to 0.4% $HNO_3$ for comparative analysis. This was repeated during the filtration experiment in order to test whether there was any instability (i.e. loss) of elements in solution in Ottawa River water which might otherwise be attributed to a loss through filtration. The unacidified sample was filtered through a subset of the filters tested previously, as follows:

| | |
|---|---|
| ***Syringe-type*** | A 5-ml aliquot of Ottawa River water was filtered and discarded prior to filtering a 50-ml aliquot which was then preserved for the subsequent determination of the 17 elements of interest. (Hg was again treated separately). |
| ***In-line-type*** | The filter was rinsed with 1-l of Type I water and then 50 to 100 ml of the sample were filtered and discarded before collecting 100 ml for analysis. Care was taken to negate dilution of the sample by pushing air through the filter (with a syringe) after rinsing. |

***Vacuum-type*** The filter (M-1-V and M-2-V) was rinsed with 100 ml of Type I water, followed by 10 ml of sample and finally a 100 ml aliquot of Ottawa River water was processed for analysis.

Each filter was tested in triplicate (i.e. repeated with new filter). All filtering was carried out within 2.5 hours and analysis was performed the same day using the procedures described previously.

In order to investigate whether any low recoveries might be due to absorption by the filter of ionic species, the experiment was repeated, again in triplicate, using spiked Type I water as the test medium. The bulk water sample was spiked at: 25 ppb in Al; 50 ppb in Fe; 120 ppt in Se; 50 ppt in Hg; and 10 ppb in all other elements. The test for Hg was performed completely separately. The unfiltered test sample was analysed at fixed periods throughout the test to monitor for any loss of analyte due to instability over that time period.

## 4.2. Results for the 15-element suite

Results for all elements are attached in Appendix 4.1. Note that data labelled as Ottawa River water alone are at fixed intervals throughout the dataset: these pertain to the unfiltered acidified portion taken periodically as the filtering experiment progressed. It is clear that the concentration of elements in the unfiltered samples of Ottawa River water did not decrease during the filtration experiment and therefore any loss can be attributed to the filtration step. The first set of data for Ottawa River water has been used for comparison, to indicate a maximum value which might be expected if all the analyte were in the 'dissolved' form. The values for the mean and standard deviation of the filtration results from Appendix 4.1 are given in Table 4.1 and are compared with the average value for the unfiltered sample.

Except for instances of contamination, results through the different filter systems for the elements Cu, As, Mo, Ag, Cd, Sb and Tl agree well with those for unfiltered Ottawa River water. Note the excellent reproducibility of most of the data, better than ±5% at levels a decade above detection limits. Results for Cu (1.44 ± 0.06 ppb) using G-1-L are significantly higher than those for unfiltered Ottawa River water (1.24 ± 0.01 ppb). This system showed a contamination level of 0.24±0.11 ppb for the first water rinse previously (Table 3.3). Arsenic and Sb results for G-2-L, the AquaPrep Thermopor system, are high (839 and 91 ppt, respectively; cf 801 and 70 ppt in the unfiltered sample), even though the filter was prerinsed with 1-l of water. This filter showed elevated concentrations of Sb previously (Table 3.3), though those for As were low and acceptable.

Elements which show considerably lower concentrations than in the unfiltered Ottawa River water sample comprise Al, Cr, Fe, Mn, Co, Zn and Pb. This probably indicates that there is a significant fraction of the element in a particulate form which is measured by ICP-MS (acidification could bring more particulate matter into solution). However, the percentage attributable to the 'dissolved' component *is not constant* and *depends upon* the filter employed, a finding in agreement with previous studies by Hall *et al.* (1996b) and Horowitz *et al.* (1996). The range in recoveries compared to the unfiltered sample are as follows:

Al 33%, for G-1-L and G-3-L to 72%, for M-1-S, M-2-S and M-1-V
Cr 67%, for G-3-L to 78%, for M-1-S and G-2-L
Fe 48%, for G-1-L and G-3-L to 79%, for M-1-S, M-2-S and M-1-V
Mn 30%, for G-1-L and G-3-L to 39%, for M-1-S, M-2-S and M-1-V
Co 40%, for G-1-L and G-3-L to 77%, for M-1-S
Zn 56%, for G-3-S to 130%, for G-1-L
Pb 45%, for G-3-L to 69%, for M-1-V

Thus, the filters ( M-1-S, M-2-S, M-1-V) made of Durapore (polyvinylidene fluoride) result in the highest recoveries of these elements, suggesting that the reproducible values of 80, 80 and 3.5 ppb, for Al, Fe and Mn, respectively, represent the <0.45 μm component. Assuming results by these three filters to be correct, the following filters produce significantly low concentrations of: Al, Fe, Mn, Co, Zn and Pb using G-1-S, G-3-S and M-4-S; Al, Fe, Mn and Co using G-1-L, G-2-L and G-3-L; and Al and Mn using M-2-V. The low result obtained for Ni by M-1-V (490 ± 14 ppt) compared to those by M-1-S and M-2-S (600 ± 14 and 563 ± 56 ppt) should be confirmed in another experiment; data for Ni are rather noisy, probably due to the correction for molecular interference from CaO species. Results for Cr, though significantly lower than that for Cr in the unfiltered sample, agree within the limits of standard deviations across all filter systems.

With the exception of Pb and Zn, results for G-1-L and G-3-L are in agreement. Both filters are made of Supor (polyethersulphone) but differ in their surface area, G-1-L being advertised as "fast-flow" presumably because of its 600 $cm^2$ surface area (cf 250 $cm^2$ for G-3-L). However, it appears that retention of elements such as Al, Fe and Mn depends not on surface area but rather on membrane material; both filters would have the same volume of sample filtered. The exceptionally low result for Pb, 69 ± 7 ppt, for G-3-L, compared to that of 96 ± 7 ppt for G-1-L, is difficult to explain. The results for Zn using these two systems - 1256 ± 89 ppt for G-1-L and 872 ± 200 ppt for G-3-L - are less reproducible and considerably higher than the 670-688 ppt average values obtained using the M-1-S, M-2-S and M-1-V systems. These higher results are probably caused by contamination as G-1-L and G-3-L were found to contribute Zn when filtering Type I water or weak acid solution (Table 3.3), the former generating a greater degree of contamination as is the case here with Ottawa River water.

Recoveries for the spiked water test samples are attached in Appendix 4.2 and are summarised in Table 4.2. Analysis of the unfiltered spiked sample throughout the experiment confirmed that there was no loss of analyte during that period. For the 15-element suite, recoveries are better than 90% except for Cr by M-1-V and Tl by M-2-V. These recoveries of 87 and 80%, respectively, are inconsistent with the earlier findings and are currently being re-assessed. The low results for elements such as Al, Fe and Mn in Ottawa River water filtered using G-1-L, G-3-L and G-3-S are not evident here for the spiked water solution. This dataset confirms that such low recoveries found for Ottawa River water pertain to problems with species other than simple ions.

## 4.3. Results for Se and Hg

Results for Se and Hg for the filtration of Ottawa River water and the spiked test water sample are attached in Appendices 4.1 and 4.2 and are summarised in Table 4.3. These experiments were carried out completely separately for Hg which was preserved immediately after filtering by making the solution 0.5% in BrCl.

Prereduction (heating in 6 M HCl) of Se VI to the reactive form, Se IV, was carried out for the Ottawa River sample but not for the Type I water which had been spiked with ca 120 ppt of Se IV. There was no loss of Se in unfiltered Ottawa River water during the experiment and therefore the initial value of 85 ± 1 ppt was used for comparison. Generally, recoveries after filtration agree with this figure (Table 4.3) but there is a tendency for slightly low results for filters G-1-L (79 ± 1 ppt), G-2-L (80 ± 2 ppt) and G-3-L (81 ± 1 ppt). Selenium is maintained in the unfiltered spiked water sample throughout the experiment, at 118 ± 2 ppt. Selenium in the filtered water samples ranges from a mean of 112 ppt to 119 ppt, small differences but still reflecting the tendency for highest recovery by M-1-S and lowest by G-1-L.

As Ottawa River water contains less than 5 ppt Hg, it was necessary to spike the unfiltered sample with ca 50 ppt (as $Hg^{2+}$). The initial mean value of 55 ± 1 ppt Hg in the unfiltered sample was used for comparison as Hg, like Se, was stable in this spiked sample of Ottawa River water during the course of the filtering experiment. The highest and lowest recoveries are attributable to filters M-1-S and M-4-S, respectively, but the differences are marked for Hg, with mean values of 44 and 13 ppt (Table 4.3). The G-1-L and G-3-L filters are again demonstrating a high degree of retention of analyte, with recoveries of only 35%. Millipore's Millex nylon and Gelman's Supor membranes are particularly absorbent for Hg.

Unlike the situation for Se, Hg was not stable in the spiked water sample during the experiment (several hours). In fact, upon shaking the sample after spiking (at 50 ppt) to attain homogeneity, the concentration of Hg was determined to be only 37 ppt. The concentration of the unfiltered sample fell continuously to a final value of 24 ppt at the end of the study. The recoveries of Hg in the filtered water samples, shown in parentheses in Table 4.3, are calculated using the value determined for the unfiltered sample closest in time (Appendix 4.2). These recoveries range from 69% for G-1-L to 91% for M-1-V, much higher levels than for Ottawa River water but still significantly low. The order of recovery of the various filters differs slightly for the two spiked samples, suggesting that the retention phenomenon is matrix-dependent, as was found for other elements.

### *Recommendations*

! Although the Acrodisc Supor membrane from Gelman was recommended for its low contamination level, recoveries of certain analytes in Ottawa River water are significantly lower than those obtained using the Millipore systems which incorporate the Durapore membrane. This indicates partial retention of the elements Al, Cr, Fe, Mn, Co, Zn and Pb present in colloidal form.

The high and consistent recoveries for all 17 elements found in the Ottawa River control with the Millipore systems advocate recommendation of the Sterivex capsule system or Durapore-based alternatives, if the goal is to measure that fraction of an element present at ≤0.45 µm. Lowest recoveries, and hence maximum retention of colloidal species, were found with Gelman's Supor membrane-based systems.

! Further assessment of the sorption of Hg, in its free ion form, by different filter systems is required. Unlike the other 16 elements, spikes of Hg added to deionised water samples were not fully recovered through all filter systems. Minimum loss is evident using the Millipore systems, particularly the Millicup bottle top model with Durapore membrane.

# 5. Stability study

The four samples of different matrix used to verify that preservation of the 16 analytes (i.e. all except Hg) in a medium of 0.4% $HNO_3$ is adequate for prolonged storage comprise Ottawa, Rideau and Gatineau River waters and a synthetic water sample which was spiked at 50 ppt in Se, 40 ppb in Al, 80 ppb in Fe and 10 ppb in the other 13 elements. Major element concentrations of these river samples are given in Table 5.1. The bulk river samples were filtered at 0.45 μm using the M-2-V system and acidified to 0.4% $HNO_3$. As their natural concentrations of Ag, Cd, Co, Sb and Tl would be too low to monitor stability with accuracy and precision, these elements were added to the three river samples to an approximate final concentration of 200-300 ppt. They were then poured into three bottles of each type (FEP, HDPE, PP and PETG) and the 48 solutions analysed at day 0 (i.e. immediately), 1, 7, 14, 21 and 28. The standard reference water sample from the National Research Council (NRC), SLRS-3, was analysed in triplicate with these batches to test for accuracy. Prior to analysis, Se was prereduced in 6 M HCl as described before.

A separate study was carried out for Hg wherein a spike equivalent to ca 50 ppt was added to each of the three filtered river samples and to a deionised water sample. Three bottle types were tested: FEP, HDPE and PETG. Each sample was preserved immediately in three different ways, in: 0.5% BrCl; 2% HCl; and 0.04% $K_2Cr_2O_7$ in 1% $HNO_3$. The experiment was performed in triplicate as above, making a total of 27 bottles per sample (Ottawa, Gatineau and Rideau River waters, and deionised water), or 108 solutions, to be analysed at day 0, 1, 7, 14, 21 and 28.

The mean and standard deviation for all 17 analytes are given in Appendix 5.1 and the data summarised in histogram form in Figure 5.1 for all elements but Hg. The only significant downward trend in concentration with time is that of Ag in the Ottawa and Gatineau River waters, from 206 ± 2 and 216 ± 1 ppt (in HDPE) to 124 ± 17 and 136 ± 5 ppt, respectively, at day 28. This is evident in each of the four bottle types. Rideau River water also shows this trend but to a lesser degree (180 ± 2 to 141 ± 3 ppt). No loss was apparent in the synthetic sample, spiked at a relatively high concentration of 10 ppb Ag. The loss of Cr in Rideau River water is not real: it is due to a molecular interference from $ArC^+$ at m/z 52. Rideau River water has a high alkalinity of 124 ppm $CaCO_3$ and a pH of 7.9. Acidification to 0.4% $HNO_3$ causes $CO_2$ to be released over several days; after a week the positive interference has gone. Ottawa River water shows this interference to a lesser degree (13 ppm $CaCO_3$, 6 ppm TOC). Results for SLRS-3 agree well with the recommended value of 0.30 ± 0.04 ppb. Alternative isotopes for Cr, of much less abundance than 52, have their own interferences. Cobalt in Rideau River water also appears to decrease, from about 0.33 to 0.22 ppb during the first week, irrespective of container. However, significant correction for CaO species on m/z 59 (only isotope of Co) is necessary for this sample and the noise caused by this correction may be misleading.

Many elements (e.g. Pb, Tl, Sb, Zn, Cu) show slightly low results for Ottawa River water at day 7 and Rideau River water at day 14, a trend which is sometimes reflected in the data for SLRS-3 (e.g. Pb and Sb in Ottawa River water). This pattern is perplexing as the obvious error due to change in calibration must be ruled out as only one sample is affected on a certain day. Furthermore, this behaviour is emulated by all bottle types.

There is a slight increase in Se concentrations in the three natural samples during the initial several days. For example, Se in Ottawa and Rideau River waters increases from 51 and 77 ppt to 70 and 95 ppt, respectively, within the first week. The synthetic sample averages 53 ppt Se at day 0 and 56 ppt at day 7, not a significant change. This increase in the river samples could correspond to an $HNO_3$ - induced liberation of a Se-organic complex, these samples containing about 5 ppm organic C.

Results for the different bottle types agree well: there is no sorption evident by any of the materials.

The stability of Hg, preserved in 0.5% BrCl in the four spiked samples contained in three different types of bottle, is excellent (Appendix 5.1). Its concentration in these samples ranges from 51 to 59 ppt over the 28-day period. Preservation in 2% HCl or 0.04% $K_2Cr_2O_7$ , however, shows a slight decrease during the first week. This is illustrated in Figures 5.2-1 and 5.2-2 for Ottawa River water. After a month, Hg in the BrCl-preserved sample remains at 55 ppt whereas Hg in the other two has stabilised at 42-43 ppt, a drop of about 20%. Precision of measurement across the triplicate series of each sample is excellent, at 2-3% RSD. Figures 5.2-2 and 5.2-3 for Ottawa River and Rideau River waters, respectively, indicate that this decrease is independent of container material (FEP, HDPE or PETG). The slightly lower initial value for Hg measured in the $K_2Cr_2O_7$ -preserved samples (51-52 ppt, cf 53-56 ppt in other two sets) may be an artifact of the analysis such as an over-correction for Hg in the blank reagent. Mercury in the Gatineau River water decreases significantly in the HCl-preserved sample (from 56 to 46 ppt) but decline in the $K_2Cr_2O_7$ -preserved counterpart is barely perceptible (Figure 5.2-4). This apparent decline in Hg may be due to a partial species transformation upon spiking, from $Hg^{2+}$ to an organic complex (especially in Ottawa and Rideau River waters) which might occur in the HCl- and $K_2Cr_2O_7$ -preserved samples, BrCl providing a stronger oxidising environment to maintain the free ion in solution and hence reactive to form $Hg^o$.

### *Recommendations*

! Acidification to 0.4% in $HNO_3$ should maintain elements Al, As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl and Zn in water samples for at least a month at room temperature. Stability over this period for Ag at concentrations of several hundred ppt is questionable and matrix-dependent. Stability of these elements is independent of container material.

! The best preservation reagent for Hg is 0.5% BrCl: it maintains Hg at ppt levels in solution for at least one month. Preservation in 2% HCl or 0.04% $K_2Cr_2O_7$ may be inadequate, especially for the former medium.

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**Table 1.1.**

Concentrations (mean±SD) of elements found in 10 ml of 0.4% $HNO_3$ placed in Fisherbrand test-tubes treated in different ways; values in ppt (ng $l^{-1}$) except for * (e.g. Al and Fe) where they are in ppb. N=50 for uncleaned ('as is') test-tubes and 125 for those soaked in 1% $HNO_3$ U U U U

| Treatment | Al* | Cr | Fe* | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| As is, uncapped | 0.14±0.13 | 11±5 | 0.2±0.8 | 1±1 | 3±4 | 2±5 | 3±6 | 67±101 | -1±2 | -5±2 | -1±<1 | <1±<1 | 2±1 | <1±<1 | 4±11 |
| As is, capped | 5.8±1.1 | 8±5 | 0.1±0.7 | 5±5 | 7±5 | <1±5 | 14±18 | 1.0±.4* | -3±2 | -3±1 | -1±<1 | <1±1 | 2±2 | <1±<1 | 2±3 |
| 1% $HNO_3$ wash | -0.1±0.01 | -1±5 | 0.2±.5 | -1±1 | 0 | -4±8 | -16±3 | -36±5 | -1±2 | -3±1 | -1±<1 | -1±1 | 1±1 | <1±<1 | -2±1 |
| Detection limit[1] | 0.03 | 10 | 1.7 | 5 | 3 | 20 | 10 | 20 | 6 | 3 | 2 | 2 | 3 | 0.2 | 2 |

[1] This instrumental detection limit is based on the equivalent of 3σ of a blank 0.4% $HNO_3$ solution

**Table 2.1.**

Mean and standard deviation (n=5) of element concentrations found in 125 ml acidified water samples contained in different bottles precleaned in different ways; all data in ppt (ng $l^{-1}$) except Fe and those with * which are in ppb. Data have not been truncated at detection limits (DL). A represents a bottle filled with 0.4% $HNO_3$ and B represents an acid-charged bottle.

| Treatment | Al | Cr | Fe* | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3σ DL | 30 | 10 | 1.7 | 5 | 3 | 20 | 10 | 20 | 6 | 3 | 2 | 2 | 3 | 0.2 | 2 |
| **FEP bottles** | | | | | | | | | | | | | | | |
| None, A | 52±63 | 67±32 | 1.9±1.1 | 13±4 | 30±12 | 492±501 | 190±199 | 98±102 | -0.7±2 | 9±4 | -0.1±0.4 | 1±1 | 2±1 | 0.09±0.1 | 10±11 |
| None, B | 176±107 | 218±117 | 42±60 | 162±201 | 69±10 | 507±54 | 1.6±2* | 766±811 | 5±7 | 164±254 | 1±1 | 3±1 | 3±2 | 0.2±0.1 | 64±59 |
| EPA, A | -10±7 | 44±24 | 0.52±0.89 | 8±6 | 17±4 | 126±24 | 34±25 | 156±330 | -1±3 | 4±1 | -0.7±0.4 | 0.3±0.3 | 0.8±0.4 | 0.1±0.05 | 1±0.4 |
| EPA, B | -2±22 | 74±16 | 1.1±1.4 | 10±5 | 40±12 | 245±39 | 31±18 | 12±7 | 0.2±1 | 16±2 | -0.7±0.3 | 1±.7 | 0.3±0.6 | 0.1±0.06 | 2±2 |
| $HNO_3$, A | -40±12 | -5±5 | 0.46±0.32 | -4±1 | 6±5 | -0.7±8 | 3±3 | 16±3 | -0.8±2 | -0.3±1 | -1±0.2 | -1±0.2 | 0.8±0.8 | 0.1±0.07 | 1±0.2 |
| $HNO_3$, B | -16±31 | 58±13 | 1.8±.8 | 5±3 | 42±11 | 233±45 | 40±24 | 34±32 | -1±1 | 20±2 | -1±0.2 | -1±0.2 | -0.2±0.3 | 0.04±0.1 | 1±0.2 |
| **HDPE bottles** | | | | | | | | | | | | | | | |
| None, A | -17±15 | 0.8±4 | 0.64±0.67 | -2±0.8 | -0.5±0.4 | -6±2 | 20±7 | 7±4 | -0.9±2 | -0.3±1 | 0.5±1 | -0.5±0.2 | 1±.5 | 0.1±0.07 | 0.8±0.8 |
| None, B | 281±19 | 4±5 | 0.61±0.91 | -2±0.6 | -0.1±0.1 | -3±3 | 32±23 | -0.6±1 | -0.7±1 | -0.4±0.7 | 0.1±0.3 | -0.1±0.5 | -0.1±0.2 | 0.1±0.07 | -0.5±0.2 |
| EPA, A | 56±29 | 16±2 | 0.29±0.50 | -3±1 | -0.4±0.3 | -9±6 | 4±1 | 12±4 | -2±1 | -0.1±0.6 | -0.7±0.3 | 0.08±0.5 | 0.6±0.2 | 0.1±0.08 | 0.9±0.6 |
| EPA, B | 312±24 | 20±4 | -0.5±0.4 | -3±0.3 | -0.4±0.3 | -13±3 | -4±2 | -4±9 | -0.02±2 | 0.2±0.5 | -0.4±0.5 | -0.1±0.4 | 0.3±0.1 | 0.1±0.02 | -0.5±0.4 |
| $HNO_3$, A | -13±8 | -9±3 | -0.1±0.3 | -3±0.4 | -0.4±0.1 | -15±5 | 4±3 | 30±9 | -2±2 | -0.6±0.5 | -1±0.2 | 0.07±0.5 | 1±0.4 | 0.02±0.1 | 1±.4 |
| $HNO_3$, B | 557±773 | 11±16 | 0.5±0.5 | -4±0.8 | -0.7±0.1 | -11±17 | -5±4 | -4±4 | -0.4±1 | -0.6±0.3 | -1±0.2 | -0.2±0.3 | -0.5±0.2 | 0.05±0.05 | -0.9±0.1 |

| Treatment | Al | Cr | Fe* | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **PETG bottles** | | | | | | | | | | | | | | | |
| None, A | 14±28 | 3±2 | 0.21±0.34 | 67±7 | 121±12 | -6±3 | 72±9 | 287±141 | 3±2 | 1±1 | 1±0.7 | 0.4±0.4 | 3±1 | 0.1±0.07 | 1±0.3 |
| None, B | 6±15 | 28±31 | 1.2±.9 | 5.3±1.0* | 9.1±1.9* | 41±10 | 90±22 | 138±23 | 2±2 | 1±2 | 0.4±0.4 | 0.07±0.3 | 14±1 | 0.09±0.07 | -0.5±0.1 |
| EPA, A | -41±15 | -1±5 | -0.26±0.61 | 0.1±1 | 2±1 | 4±5 | 8±2 | 43±23 | 0.2±2 | -0.5±0.4 | -0.7±0.3 | -0.04±0.6 | 0.7±0.5 | 0.07±0.08 | 0.9±0.2 |
| EPA, B | -33±7 | 2±4 | 0.29±0.68 | 2.1±.6* | 3.3± 1.1* | 8±5 | 3±1 | 126±220 | 0.02±2 | 0.6±0.8 | -0.5±0.4 | 0.2±0.4 | 7±2 | 0.1±0.02 | -0.5±0.1 |
| $HNO_3$, A | -48±4 | -4±4 | -0.39±0.53 | -1±2 | 1±2 | -11±4 | 5±3 | 9±5 | -1±0.3 | -0.4±0.6 | -0.7±0.3 | -0.2±0.3 | 0.8±1 | 0.02±0.07 | -0.5±0.1 |
| $HNO_3$, B | -54±9 | -1±3 | <0.1±0.4 | 2.7±1.0* | 4.5±1.7* | 10±9 | -1±3 | 57±90 | -1±0.3 | -0.1±0.8 | -0.9±0.4 | 0.2±0.1 | 9±3 | 0.05±0.05 | -0.7±0.2 |
| **PP bottles** | | | | | | | | | | | | | | | |
| None, A | 594±40 | -4±6 | -1±0.47 | -1±1 | -0.1±0.2 | -6±4 | 39±13 | 28±10 | 2±2 | 0.05±0.6 | 0.4±0.4 | -0.2±0.2 | 1±0.6 | 0.2±0.1 | 1±0.4 |
| None, B | 4.2±0.2* | 9±5 | -0.2±0.5 | -1±1 | -0.6±0.3 | -0.5±4 | 86±100 | 28±33 | -1±2 | -0.06±0.6 | 1±0.5 | -0.3±0.2 | 0.6±0.5 | 0.1±0.1 | 0.07±0.1 |
| EPA, A | 66±26 | 20±1 | 0.07±0.64 | -2±0.5 | -0.3±0.3 | -5±6 | 3±3 | 2±2 | 7±2 | -0.6±0.2 | -0.2±0.1 | 0.06±0.3 | 0.6±0.3 | 0.1±0.1 | 0.7±0.1 |
| EPA, B | 724±62 | 20±3 | 0.13±0.40 | -2±0.2 | -0.08±0.1 | -11±4 | -3±3 | -6±3 | -0.06±3 | -0.06±0.6 | -0.3±0.3 | 0.04±0.2 | 0.4±0.9 | 0.1±0.07 | -0.8±0.3 |
| $HNO_3$, A | -47±24 | -8±2 | -0.3±0.5 | -3±1 | -0.3±0.4 | -12±5 | 3±4 | 25±12 | 0.2±2 | -0.2±0.3 | -0.6±0.2 | 0.2±0.4 | 0.9±0.7 | 0.02±0.1 | 1±0.9 |
| $HNO_3$, B | 842±100 | 2±3 | 0.03±0.50 | -5±0.5 | -0.6±0.1 | -12±3 | -2±7 | -3±5 | -3±4 | -0.5±0.8 | -1.1±0.5 | -0.01±0.4 | -0.4±0.4 | -0.01±0.0 | -0.9±0.2 |
| **HDPE bottles, purchased as 'precleaned'** | | | | | | | | | | | | | | | |
| A | 133±48 | 35±6 | 0.4±0.5 | 2±2 | -0.1±0.2 | 17±3 | 24±5 | 739±195 | 0.2±1 | 0.8±0.8 | -0.3±0.3 | 0.5±0.5 | 0.2±0.5 | 0.1±0.1 | 4±2 |
| B | 148±41 | 102±23 | 0.8±0.6 | 5±5 | -0.07±0.3 | 22±9 | 14±8 | 2.0±1.2* | 0.1±2 | 0.3±1 | -0.3±0.4 | 0.2±0.4 | 1±0.8 | 0.06±0.1 | 4±2 |

None = rinse three times with water

EPA = Modified EPA cleaning method 1638 based on washing with soap solution followed by concentrated $HNO_3$ (no HCl step); note 5% $HNO_3$ substituted for conc. $HNO_3$ for PETG bottles which would otherwise melt

$HNO_3$ = State of Virginia cleaning method, based on washing with 5 and 0.5% $HNO_3$

**Table 2.2.**

Mean and standard deviation (n=5) values in ppt for Hg found in 125 ml bottles made of 4 different materials cleaned by three different methods and Hg preserved in three different reagents. Instrumental 3σ DL is 1 ppt.

| | Preservation method | | |
|---|---|---|---|
| Bottle material | In 0.5% BrCl | In 2% HCl | In 0.04% $K_2Cr_2O_7$ |
| | | **EPA Method 1631** | |
| FEP | -0.6 ± 0.3 | -0.1 ± 0.3 | 2.6 ± 0.5 |
| HDPE | -0.9 ± 0.2 | -1.1 ± 0.5 | 1.8 ± 0.2 |
| PETG | -0.4 ± 0.1 | -0.7 ± 0.2 | 1.5 ± 0.7 |
| PP | -0.3 ± 0.5 | -0.2 ± 0.3 | 0.6 ± 0.1 |
| | | **EPA Method 1638** | |
| FEP | -0.1 ± 0.2 | -0.1 ± 0.1 | 1.8 ± 0.4 |
| HDPE | -0.1 ± 0.1 | -0.3 ± 0.1 | 1.7 ± 0.2 |
| PETG | 0.2 ± 0.1 | 0.6 ± 0.1 | 0.9 ± 0.4 |
| PP | <0.1 | 0.2 ± 0.1 | 0.6 ± 0.1 |
| | | **No cleaning** | |
| FEP | 0.7 ± 0.4 | 0.2 ± 0.5 | 1.7 ± 0.9 |
| HDPE | -0.3 ± 0.3 | 0.4 ± 0.5 | 1.5 ± 0.4 |
| PETG | -0.5 ± 0.1 | 0.1 ± 0.3 | 1.1 ± 0.2 |
| PP | -0.1 ± 0.2 | 0.1 ± 0.4 | 1.4 ± 0.8 |
| HDPE (precleaned) | 0.4 ± 0.3 | 0.3 ± 0.2 | 1.3 ± 0.2 |

**Table 2.3.**

Element concentrations found in stream water surveys used to estimate method detection limits (MDL) required for hydrogeochemical exploration; values in ppt except where designated as * to indicate ppb.

| | Al* | Cr* | Fe* | Mn* | Co | Ni* | Cu* | Zn* | As* | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | **Nova Scotia survey of 729 streams** | | | | | | | | |
| Minimum | <2 | <0.1 | <3.5 | 1.4 | <50 | <0.2 | <0.1 | <0.5 | <0.1 | <50 | <50 | <50 | <10 | <5 | <100 |
| Mean ± SD | 142±114 | 0.2±0.2 | 277±259 | 87±177 | 234±441 | 0.34±0.88 | 0.77±2.8 | 2.6±2.4 | 0.5±2.8 | 89±326 | all <50 | 51±62 | 46±541 | 3±1.8 | 284±495 |
| | | | | | | | **Newfoundland survey of 139 streams** | | | | | | | | |
| Minimum | <10 | <0.1 | 23 | <1 | 16 | 0.4 | <0.1 | 0.4 | <0.2 | <50 | <50 | <10 | 6 | <5 | <20 |
| Mean ± SD | 207±185 | 0.2±0.2 | 274±284 | 33±83 | 306±544 | 1.2±1.1 | 2.7±5.9 | 2.7±3.7 | 0.21±0.44 | 93±171 | all <50 | 39±162 | 24±25 | 3±0.6 | 112±476 |
| MDL - geo | 0.5 | 0.05 | 1 | 0.1 | 10 | 0.05 | 0.05 | 0.1 | 0.05 | 10 | 1 | 5 | 1 | <1 | 10 |
| MDL - env | 0.5 | 0.2 | 30 | 10 | 5000 | 2 | 0.1 | 2 | 5 | 200 | 10 | 10 | 2000 | 30 | 100 |
| Criteria[1] | 5 | 20 | 300 | | 50000 | 25 | 2 | 30 | 50 | | 100 | 200 | | | 1000 |
| Criteria[2] | 5 | 2 | 300 | 50 | 900 | 25 | 2 | 14 | 5 | 10000 | 50 | 10 | 6000 | 300 | 3000 |

SD: standard deviation

MDL-geo: method detection limit required in hydrogeochemical surveys

MDL-env: method detection limit typically required by environmental regulations

Criteria[1]: lowest remediation criteria level (for irrigation, freshwater aquatic life, drinking water) required by CCME (1991)

Criteria[2]: lowest remediation criteria level required by B.C. (Larry Pommen, Victoria, pers. comm., 1998)

**Table 2.4.**

Elements are shown where the value in the filled test bottle (0.4% $HNO_3$) for the 'mean plus two standard deviations' (taken from Table 2.1) is greater than the required method detection limit in hydrogeochemical and environmental surveys. Underlined indicates elements of particular concern (i.e. high contamination levels expected).

| Cleaning - application* | FEP | HDPE | HDPE$^{P}$ | PETG | PP |
|---|---|---|---|---|---|
| None - geo | Cr, Fe, Co, Ni, Cu, Zn, Mo, Pb | | Zn | Co, Cu, Zn, Sb | Al, Cu, Ag, Sb |
| None - env | Cu | | | | Al |
| EPA - geo | Cr, Co, Ni, Cu, Zn, | | | | |
| EPA - env | | | | | |
| $HNO_3$ - geo | Co | | | | |
| $HNO_3$ - env | | | | | |

*geo- geochemical, env - environmental

**Table 3.1.**

Mean and standard deviation ($n = 3$) for water or 0.4% $HNO_3$ rinses of Becton Dickinson (BD) and Whatman (Wha) plastic syringes to test for potential contamination; values in ppt except where indicated by * where they are in ppb. (Values not truncated at analytical detection limits)

| Syringe, content | Al | Cr | Fe* | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| BD, water | 62±114 | 4±5 | -0.3±0.3 | 2±2 | -1±0.6 | 7±5 | -20±10 | 0.4±0.4* | <1±<1 | 1±0.5 | <1±<1 | <1±<1 | <1±<1 | <1±<1 | 1±1 |
| Wha, water | 23±18 | 5±5 | -0.8±4 | 1±1 | -1±<1 | 9±19 | 5±23 | 1±36 | 1±<1 | 1±<1 | <1±<1 | 1±1 | <1±<1 | <1±<1 | <1±<1 |
| BD, 0.4% $HNO_3$ | 1290±360 | 144±81 | 1.1±1.0 | 42±6 | 4±<1 | 168±45 | 75±14 | 119±36* | 1±1 | 10±13 | <1±<1 | 2±2 | 3±2 | <1±1 | 8±10 |
| Wha, 0.4% $HNO_3$ | 269±34 | 123±5 | 1.6±1.2 | 14±1 | 2±<1 | 143±2 | -4±18 | -3±22 | <1±1 | 34±52 | 1±<1 | <1±1 | 2±<1 | 1<1 | 7±7 |

**Table 3.2.**

Description of different 0.45 μm (and 2-5 μm) filter systems investigated. G - Gelman; M - Millipore; F - Falcon; S - syringe; V - vacuum; L - in-line

| Designation, part # | Description* |
|---|---|
| G-1-S, #4585 | Ion chromatography Acrodisc, Supor (polyethersulphone) |
| G-2-S, #4549 | Nylon Acrodisc GF, nylon with glass fibre prefilter |
| G-3-S, #4560 | GHP Acrodisc, hydrophilic polypropylene |
| M-1-S, #SVHV L 10 15 | Sterivex capsule, hydrophilic Durapore (polyvinylidene fluoride, PDVF) |
| M-2-S, #SLHV 025 NS | Millex HV, hydrophilic Durapore |
| M-3-S, SLLS 025 NS or SLSV | Millex-LS 5μm prefilter or Millex-SV |
| M-4-S, #SLHN 025 NS | Millex HN, nylon |
| M-1-V, #SJHV 47 10 | Millicup bottle top, Durapore membrane |
| F-1-V, #7104 | Bottle top, MF filter (gridded filter substituted by mixed cellulose esters) |
| M-2-V, #XX15 047 00 | All glass unit, MF filters (HAWP 047 00, mixed cellulose esters) |
| G-1-L, #12175 or 12176 | Groundwater sampling capsule, AquaPrep 600, Supor (polyethersulfone), designed to be fast flow (area of 600 $cm^2$) |
| G-2-L, 4270 | AquaPrep, Thermopor (polyester reinforced polysulfone) |
| G-3-L, #12026 or 12027 | AquaPrep 250, Supor membrane as G-1-L, polypropylene housing |
| M-3-V, #XX15 047 00 | As M-2-V but 5 μm filter size (SMWP 047 00) |

* Material named (e.g. Supor, Durapore) refers to membrane rather than filter housing

**Table 3.3.**

Values for the mean and standard deviation (n = 3) of water (W) or 0.4% $HNO_3$ (A) rinses of different filter systems to test for potential contamination; values in ppt except where indicated by * where they are in ppb. All except M-3-S and M-3-V (5 μm) are at the 0.45 μm pore size. (Values are not truncated at analytical detection limits)

| Filter | Al | Cr | Fe* | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| G-1-S, W | -4/7 | 7±7 | 0.06±1.2 | 14±2 | <1±<1 | 52±3 | -11±6 | -21±5 | 1±2 | 2±1 | <1±<1 | 2±1 | 1±1 | 1±<1 | <1±<1 |
| G-1-S, A | 20±12 | -12±2 | 0.42±1.4 | 3±2 | -1±<1 | 15±3 | -7±12 | 69±80 | <1±<1 | 3±3 | <1±<1 | 2±2 | 2±1 | <1±<1 | 4±1 |
| G-2-S, W | 63±10 | -3±1 | 1.5±4.4 | 15±2 | -1±<1 | 18±6 | 71±13 | 659±124 | 9±1 | 7±<1 | <1±<1 | 1±<1 | 2±2 | <1±<1 | 1±<1 |
| G-2-S, A | 26±0.2* | 37±9 | 6.0±1.0 | 78±2 | -1±<1 | 23±1 | 1.5±0.5* | 119±9* | 7±1 | 2±1 | <1±<1 | 20±1 | 5±<1 | 6±<1 | 666±33 |
| G-3-S, W | 56±49 | -13±13 | 0.2±1.1 | 17±3 | <1±<1 | 44±10 | <1±5 | 15±20 | -2±1 | <1±1 | <1±<1 | 2±4 | 1±<1 | <1±<1 | 2±1 |
| G-3-S, A | 52±10 | -6±4 | 3.5±0.8 | 21±1 | -1±<1 | 10±2 | -8±9 | 65±101 | -2±2 | <1±1 | <1±<1 | 2±2 | 2±1 | <1±<1 | 4±1 |
| M-1-S, W | 196±128 | -8±18 | -1.0±1.6 | 7±5 | 5±3 | 8±3 | -17±8 | 28±30 | -1±2 | 2±2 | <1±<1 | -1±1 | <1±2 | <1±<1 | 1±<1 |
| M-I-S, A | 81±35 | 7±8 | -1.3±2.1 | 9±1 | 9±1 | 7±3 | 71±123 | 99±25 | -1±1 | 1±1 | <1±<1 | <1±1 | 2±<1 | <1±<1 | 10±4 |
| M-2-S, W | 11±15 | -2±13 | 1.1±0.9 | 1±1 | -1±<1 | 4±4 | 56±117 | -21±1 | <1±1 | 2±4 | <1±<1 | <1±1 | <1±1 | <1±<1 | 3±5 |
| M-2-S, A | 1.2±2.0* | -4±5 | 0.3±1.4 | 2±1 | -1±<1 | 16±6 | 12±38 | 160±159 | -1±1 | <1±1 | <1±1 | 2±2 | 1±1 | <1±<1 | 7±6 |
| M-3-S, W | -1±11 | 2±3 | 1.1±1.7 | 2±<1 | -1±<1 | 8±<1 | -13±9 | 12±52 | <1±1 | 1±1 | <1±<1 | 1±1 | 1±1 | <1±<1 | 4±8 |
| M-3-S, A | 22±12 | 4±4 | 2.7±1.7 | 2±1 | -1±<1 | 13±4 | 13±5 | 101±93 | 1±2 | 1±<1 | -1±<1 | 2±2 | 4±1 | <1±<1 | 3±1 |
| M-4-S, W | 7±17 | 7±9 | 3.2±3.1 | 1±1 | -1±<1 | 10±2 | -8±7 | 36±51 | -1±1 | <1±<1 | -1±<1 | 1±1 | <1±<1 | <1±<1 | 1±<1 |
| M-4-S, A | 46±39 | -1±9 | 2.7±1.2 | 1±<1 | <1±2 | 13±6 | -2±20 | 30±55 | 1±<1 | 2±2 | <1±<1 | 1±1 | <1±1 | <1±<1 | 6±6 |
| M-1-V, W1 | 64±37 | 37±51 | 1.7±1.3 | 7±8 | 3±5 | 35±46 | -2±2 | 44±52 | 1±<1 | 3±4 | <1±<1 | 1±1 | 1±1 | <1±<1 | 3±5 |
| M-1-V, W2 | 4±12 | 8±4 | 4.2±1.7 | 1±1 | -1±<1 | 4±<1 | -8±14 | -3±10 | 1±1 | <1±1 | <1±<1 | 1±1 | <1±<1 | <1±<1 | <1±<1 |
| M-1-V, A | 134±49 | 15±7 | 2.9±0.7 | 4±1 | -1±1 | 16±7 | 31±13 | 385±370 | 2±1 | <1±<1 | <1±<1 | 2±1 | 1±<1 | <1±<1 | 15±1 |
| F-1-V, W1 | 118±140 | 18±4 | 1.1±2.3 | 49±6 | -1±<1 | 4±3 | 28±27 | 1.4±0.04* | 3±1 | 3±1 | <1±<1 | 2±1 | <1±1 | <1±<1 | 29±46 |
| F-1-V, W2 | -9±5 | 2±8 | 3.4±1.3 | 3±1 | -1±<1 | 3±2 | -8±14 | 163±32 | 1±<1 | <1±<1 | <1±<1 | <1±2 | 1±1 | <1±<1 | 2±3 |

| Filter | Al | Cr | Fe* | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| F-1-V, A | 86±46 | <1±11 | 0.4±2.6 | 8±1 | -1±<1 | 2±4 | -3±15 | 511±91 | <1±1 | -1±<1 | <1±<1 | 1±1 | 1±<1 | <1±<1 | 5±1 |
| M-2-V, W1 | 335±262 | 1±3 | 0.7±2.6 | 4±2 | <1±1 | -6±23 | 18±9 | 116±83 | <1±3 | -1±1 | -1±1 | 3±2 | 2±1 | <1±<1 | 12±16 |
| M-2-V, W2 | 249±227 | -2±4 | -2.2±2.5 | 1±2 | <1±<1 | -2±10 | 51±36 | 97±26 | -1±2 | -1±1 | <1±<1 | 1±2 | 1±1 | <1±<1 | 17±20 |
| M-2-V, A | 890±39 | -4±4 | -2.2±1.4 | 13±3 | <1±<1 | 6±11 | 398±90 | 1.1±0.2* | -4±4 | -2±1 | <1±<1 | 4±1 | 2±1 | <1±<1 | 91±26 |
| G-1-L, W1 | 578±40 | 74±35 | 1.6±1.9 | 30±8 | 1±<1 | 92±39 | 240±110 | 379±211 | 8±4 | 17±5 | <1±<1 | 3±2 | 3±2 | <1±<1 | 16±3 |
| G-1-L, W2 | 46±33 | 27±13 | 2.7±2.9 | 4±3 | -1±<1 | 7±3 | -14±7 | 6±13 | <1±<1 | <1±1 | <1±<1 | 1±1 | 1±1 | <1±<1 | <1±<1 |
| G-1-L, A | 1.2±0.1* | 35±6 | 3.4±2.5 | 34±4 | 1±<1 | 98±10 | 620±82 | 1.8±0.5* | 1±<1 | 1±1 | <1±<1 | 8±3 | 7±1 | <1±<1 | 165±49 |
| G-2-L, W1 | -13±10 | 7±13 | -3.6±3.1 | 183±21 | 86±16 | 10±4 | -18±4 | 14±19 | 2±1 | <1±1 | <1±<1 | 1±<1 | 292±19 | <1±<1 | 4±<1 |
| G-2-L, W2 | -12±5 | 11±3 | 0.1±1.0 | 9±1 | 2±1 | 8±5 | -13±15 | -12±19 | 3±1 | 1±1 | <1±<1 | <1±<1 | 34±7 | <1±<1 | 2±1 |
| G-2-L, A | 77±55 | 3±4 | 1.0±2.5 | 31±4 | 8±2 | 9±1 | -12±8 | 53±46 | 2±<1 | <1±1 | <1±<1 | 1±1 | 231±11 | <1±<1 | 58±18 |
| G-3-L, W1 | 124±21 | 8±12 | 1.4±2.1 | 11±4 | <1±<1 | 38±10 | 10±5 | 112±2 | 5±2 | 8±1 | <1±<1 | 2±<1 | 1±<1 | <1±<1 | <1±<1 |
| G-3-L, W2 | 23±15 | -2±5 | 0.3±0.3 | 1±<1 | -1±<1 | 5±3 | -18±2 | -14±3 | 1±1 | <1±2 | <1±1 | <1±1 | <1±<1 | <1±<1 | <1±<1 |
| G-3-L, A | 1.0±0.1* | 7±9 | 2.9±1.0 | 77±36 | 1±1 | 62±15 | 549±55 | 2.0±0.2* | 2±1 | 5±1 | 1±1 | 10±5 | 4±<1 | <1±<1 | 23±6 |
| M-3-V, W1 | 44 | 3 | 1.9 | 2 | -1 | 12 | 33 | 16 | <1 | <1 | <1 | <1 | <1 | <1 | 5 |
| M-3-V, W2 | 260 | 3 | 2.7 | 1 | -1 | 12 | -3 | 2 | -1 | <1 | <1 | <1 | <1 | <1 | 8 |
| M-3-V, A | 465 | 8 | 2.3 | 8 | <1 | 29 | 269 | 652 | -2 | -1 | <1 | 11 | <1 | <1 | 206 |

G - Gelman; M - Millipore; F - Falcon; S - syringe; L - in-line; V - vacuum; W - water sample (W1 = first pass; W2 = after rinsing with 300 or 900 ml); A - acidic sample

**Table 3.4.**

List of elements where contamination is evident for filter systems tested, with respect to applications in geochemistry and environmental projects.

| | Water sample | | Acidic sample |
|---|---|---|---|
| Filter system | Geochemistry | Environmental | Environmental |
| G-1-S | Ni | | |
| G-2-S | Cu, Zn | | Al, Cu, Zn, Cd, Pb |
| G-3-S | Ni, Cd | Cd | |
| M-1-S | Co | | Cu |
| M-2-S | Cu, Mo, Pb | Cu | Al |
| M-3-S | Zn, Pb | | |
| M-4-S | Fe, Zn | | |
| M-1-V | Cr, Co, Ni, Zn, Mo, Pb | | |
| F-1-V | Cu, Zn, Pb | Pb | |
| M-2-V | Al, Cu, Zn, Pb | Al, Cu | Al, Cu, Pb |
| G-1-L | Al, Cr, Ni, Cu, Zn, Mo, Cd, Sb, Pb | Al, Cu | Al, Cu, Zn, Pb |
| G-2-L | Mn, Co, Sb | | |
| G-3-L | Ni, Zn, Mo | | Al, Cu, Zn, Cd |
| M-3-V* | | | Cu, Pb |

* difficult to assess as this 5 μm system was analysed only once, same material as M-2-V

**Table 4.1.**

Values for mean and standard deviation (n=3) for elements in Ottawa River water filtered through different 0.45 µm systems, compared to unfiltered acidified Ottawa River water. Results in ppt unless designated by * where they are in ppb.

| Filter | Al* | Cr | Fe* | Mn* | Co | Ni | Cu* | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Unfiltered | 111±4.5 | 495±6 | 104±3.7 | 8.9±0.14 | 35±1 | 581±6 | 1.24±0.01 | 959±1 | 801±8 | 258±1 | 2±1 | 15±1 | 70±2 | 6±1 | 152±2 |
| G-1-S | 56±2.6 | 364±8 | 65±3.4 | 3.1±0.02 | 16±1 | 515±14 | 1.18±0.02 | 548±20 | 766±15 | 257±1 | 1±1 | 12±1 | 67±1 | 6±1 | 84±1 |
| G-3-S | 42±1.5 | 346±6 | 63±1.8 | 2.8±0.05 | 15±1 | 502±75 | 1.20±0.03 | 536±4 | 774±9 | 254±9 | 1±0 | 12±2 | 69±2 | 5±1 | 75±2 |
| M-1-S | 80±1.1 | 387±13 | 82±1.9 | 3.5±0.04 | 27±1 | 600±14 | 1.23±0.00 | 675±16 | 785±4 | 257±1 | 1±0 | 11±1 | 70±1 | 6±1 | 102±2 |
| M-2-S | 80±0.9 | 366±8 | 77±5.2 | 3.5±0.03 | 22±1 | 563±56 | 1.20±0.03 | 670±14 | 778±10 | 254±6 | 2±1 | 13±2 | 70±2 | 7±1 | 99±1 |
| M-4-S | 58±1.0 | 353±9 | 69±1.8 | 3.0±0.03 | 17±1 | 543±6 | 1.20±0.01 | 599±18 | 783±5 | 254±2 | 1±0 | 11±1 | 70±2 | 6±0 | 86±2 |
| G-1-L | 37±2.1 | 364±22 | 51±5.2 | 2.7±0.19 | 14±1 | 502±51 | 1.44±0.06 | 1256±89 | 784±51 | 253±10 | 1±0 | 14±1 | 71±3 | 6±2 | 96±7 |
| G-2-L | 58±1.7 | 387±12 | 67±1.8 | 3.2±0.03 | 18±1 | 562±19 | 1.21±0.09 | 633±20 | 839±18 | 263±4 | 2±1 | 11±1 | 91±6 | 6±1 | 97±2 |
| G-3-L | 38±0.5 | 332±26 | 50±2.9 | 2.6±0.06 | 14±1 | 507±70 | 1.35±0.18 | 872±200 | 780±11 | 257±6 | 1±1 | 12±1 | 71±2 | 6±1 | 69±7 |
| M-1-V | 80±1.3 | 363±8 | 79±0.7 | 3.5±0.06 | 21±1 | 490±14 | 1.24±0.02 | 688±19 | 799±8 | 263±1 | 1±1 | 14±1 | 70±1 | 7±1 | 105±1 |
| M-2-V | 67±1.9 | 365±17 | 76±1.6 | 3.1±0.03 | 20±1 | 560±26 | 1.25±0.01 | 712±45 | 804±8 | 263±6 | 2±1 | 16±3 | 74±2 | 5±1 | 99±5 |

**Table 4.2.**

Values for the mean and standard deviation (n=3) of elements in spiked deionised water sample, with different filters; values in ppb.

| Filter | Al | Cr | Fe | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| G-1-S | 25±0.2 | 10.0±0.1 | 49.3±0.5 | 10.0±0.2 | 9.9±0.1 | 10.0±0.1 | 9.9±0.1 | 10.0±0.1 | 10.0±0.1 | 9.9±0.1 | 10.0±0.1 | 9.9±0.1 | 9.9±0.2 | 9.9±0.1 | 9.9±0.1 |
| G-3-S | 25±0.3 | 10.2±0.2 | 48.7±0.7 | 10.0±0.2 | 10.1±0.1 | 10.2±0.1 | 10.2±0.2 | 10.2±0.1 | 10.0±0.2 | 10.0±0.1 | 10.0±0.1 | 9.9±0.1 | 9.8±0.1 | 10.0±0.0 | 10.0±0.0 |
| M-1-S | 25±0.2 | 10.1±0.1 | 46.8±0.6 | 10.1±0.1 | 10.0±0.1 | 10.2±0.1 | 10.0±0.2 | 10.3±0.1 | 10.0±0.0 | 10.0±0.0 | 10.0±0.1 | 10.0±0.1 | 10.0±0.3 | 10.0±0.1 | 10.1±0.1 |
| M-2-S | 26±0.2 | 10.2±0.1 | 49.3±0.7 | 10.0±0.1 | 10.1±0.0 | 10.2±0.1 | 10.1±0.1 | 10.2±0.1 | 10.0±0.1 | 10.0±0.1 | 10.1±0.0 | 10.0±0.1 | 10.0±0.1 | 10.0±0.0 | 10.0±0.0 |
| M-4-S | 25±0.4 | 10.0±0.1 | 50.0±0.9 | 10.0±0.1 | 10.1±0.1 | 10.1±0.1 | 10.1±0.1 | 10.1±0.2 | 9.9±0.1 | 10.0±0.1 | 10.0±0.1 | 9.9±0.1 | 9.7±0.4 | 10.0±0.0 | 10.0±0.0 |
| G-1-L | 25±0.4 | 9.9±0.1 | 49.4±1.3 | 9.9±0.1 | 10.0±0.0 | 10.0±0.1 | 10.2±0.1 | 10.4±0.3 | 9.8±0.1 | 9.9±0.1 | 10.0±0.1 | 9.9±0.1 | 9.8±0.1 | 9.9±0.1 | 9.9±0.0 |
| G-2-L | 25±0.2 | 9.9±0.1 | 51.8±0.3 | 10.0±0.1 | 10.0±0.1 | 10.0±0.1 | 10.0±0.2 | 9.9±0.1 | 9.9±0.1 | 10.0±0.0 | 9.9±0.0 | 9.9±0.1 | 9.9±0.4 | 9.9±0.1 | 10.0±0.1 |
| G-3-L | 25±0.6 | 9.9±0.2 | 50.8±1.1 | 10.1±0.1 | 10.0±0.1 | 10.1±0.1 | 10.2±0.2 | 10.5±0.4 | 10.1±0.1 | 10.2±0.1 | 9.9±0.1 | 10.0±0.1 | 10.3±0.3 | 10.1±0.2 | 10.1±0.1 |
| M-1-V | 24±0.1 | 8.6±0.2 | 46.2±1.5 | 9.8±0.2 | 9.8±0.2 | 9.8±0.1 | 9.8±0.2 | 9.9±0.2 | 9.8±0.1 | 9.9±0.2 | 9.9±0.1 | 9.8±0.1 | 9.9±0.2 | 9.8±0.2 | 9.4±0.2 |
| M-2-V | 25±0.1 | 9.9±0.3 | 42.9±4.6 | 10.1±0.1 | 10.0±0.0 | 10.1±0.1 | 10.0±0.1 | 10.3±0.2 | 10.0±0.0 | 9.9±0.1 | 9.4±0.2 | 10.0±0.1 | 9.7±0.1 | 8.2±0.7 | 9.5±0.5 |

**Table 4.3.**

Values for the mean and standard deviation (n=3) of Se and Hg in Ottawa River water (spiked at ca 50 ppt in Hg) and a deionised water sample (spiked at ca 120 ppt Se and 50 ppt Hg), with different filters.

| | Se, ppt | | Hg, ppt | |
|---|---|---|---|---|
| Filter | Ottawa River | Spiked water | Spiked Ottawa R* | Spiked water* |
| G-1-S | 85±1 | 117±3 | 40±1 (74%) | 30±1 (83%) |
| G-3-S | 84±3 | 115±1 | 33±0 (60%) | 29±1 (81%) |
| M-1-S | 84±3 | 119±3 | 44±0 (81%) | 30±1 (84%) |
| M-2-S | 83±2 | 118±5 | 42±1 (78%) | 31±1 (87%) |
| M-4-S | 83±1 | 115±1 | 13±1 (23%) | 27±0 (76%) |
| G-1-L | 79±1 | 112±2 | 19±1 (34%) | 21±2 (69%) |
| G-2-L | 80±2 | 114±2 | 34±1 (63%) | 28±6 (87%) |
| G-3-L | 81±1 | 115±2 | 19±1 (36%) | 20±1 (73%) |
| M-1-V | 82±1 | 115±4 | 38±1 (70%) | 23±1 (91%) |
| M-2-V | 86±2 | 117±6 | 33±1 (61%) | 18±1 (76%) |

*: Recoveries compared to unfiltered sample are shown in parentheses

**Table 5.1.**

Characteristics of the Ottawa, Rideau and Gatineau River water samples.

| Sample | pH | TOC, ppm | Alkalinity* | Na, ppm | Ca, ppm | Mg, ppm | K, ppm |
|---|---|---|---|---|---|---|---|
| Ottawa | 7.1 | 5.7 | 12.6 | 1.87 | 6.5 | 1.6 | 0.65 |
| Rideau | 7.9 | 5.3 | 124 | 9.15 | 39.5 | 12.7 | 1.18 |
| Gatineau | 3.5 | <0.5 | <1.0 | 1.03 | 4.8 | 0.9 | 0.45 |

* as $CaCO_3$, in ppm

# BOTTLE TESTING

**Ag, Al, As, Cd, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Zn, Co, Tl**

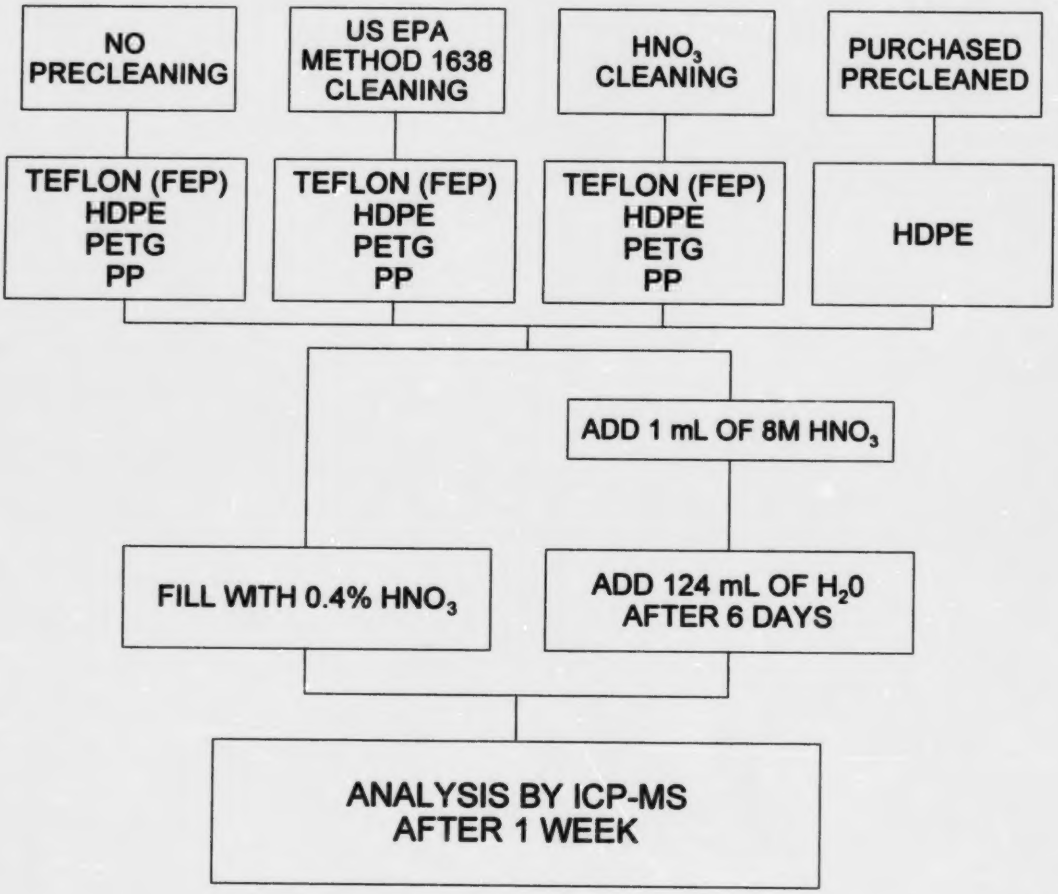

NO PRECLEANING
US EPA METHOD 1638 CLEANING
HNO3 CLEANING
PURCHASED PRECLEANED
TEFLON (FEP) HDPE PETG PP
TEFLON (FEP) HDPE PETG PP
TEFLON (FEP) HDPE PETG PP
HDPE
ADD 1 mL OF 8M HNO3
FILL WITH 0.4% HNO3
ADD 124 mL OF H20 AFTER 6 DAYS
ANALYSIS BY ICP-MS AFTER 1 WEEK

Figure 2.1

| | | | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FEP | NONE | 0.4% HNO3 | 0.053 | 0.066 | 1.666 | 0.014 | 0.03 | 0.493 | 0.19 | 0.099 | -0.001 | 0.01 | 0 | 0.001 | 0.002 | 0 | 0.01 |
| FEP | NONE | 1 mL of 8M HNO3 | 0.177 | 0.218 | 41.702 | 0.162 | 0.069 | 0.507 | 1.615 | 0.767 | 0.005 | 0.164 | 0.001 | 0.004 | 0.003 | 0 | 0.064 |
| FEP | EPA | 0.4% HNO3 | -0.011 | 0.044 | 0.522 | 0.008 | 0.018 | 0.127 | 0.034 | 0.156 | -0.001 | 0.004 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| FEP | EPA | 1 mL of 8M HNO3 | -0.002 | 0.075 | 1.156 | 0.011 | 0.04 | 0.246 | 0.032 | 0.012 | 0 | 0.017 | -0.001 | 0.001 | 0 | 0 | 0.002 |
| FEP | VIRGINIA | 0.4% HNO3 | -0.04 | -0.005 | 0.465 | -0.004 | 0.007 | 0.001 | 0.003 | 0.017 | -0.001 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| FEP | VIRGINIA | 1 mL of 8M HNO3 | -0.016 | 0.058 | 1.79 | 0.005 | 0.043 | 0.234 | 0.041 | 0.034 | -0.001 | 0.02 | -0.001 | 0.001 | 0 | 0 | 0.001 |
| | | | | | | | | | | | | | | | | | |
| HDPE | NONE | 0.4% HNO3 | -0.017 | 0.001 | 0.642 | -0.002 | -0.001 | -0.006 | 0.02 | 0.008 | 0.001 | 0 | 0.001 | -0.001 | 0.001 | 0 | 0.001 |
| HDPE | NONE | 1 mL of 8M HNO3 | 0.281 | 0.004 | 0.611 | -0.002 | 0 | -0.003 | 0.033 | -0.001 | -0.001 | 0 | 0 | 0 | 0 | 0 | 0 |
| HDPE | EPA | 0.4% HNO3 | 0.057 | 0.016 | 0.292 | -0.003 | 0 | -0.009 | 0.004 | 0.013 | -0.002 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| HDPE | EPA | 1 mL of 8M HNO3 | 0.313 | 0.02 | -0.494 | -0.003 | 0 | -0.013 | -0.005 | -0.004 | 0 | 0 | 0 | 0 | 0 | 0 | -0.001 |
| HDPE | VIRGINIA | 0.4% HNO3 | -0.014 | -0.009 | -0.126 | -0.004 | 0 | -0.015 | 0.005 | 0.03 | -0.002 | -0.001 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| HDPE | VIRGINIA | 1 mL of 8M HNO3 | 0.557 | 0.011 | 0.565 | -0.004 | -0.001 | -0.012 | -0.005 | -0.004 | 0 | -0.001 | -0.001 | 0 | 0 | 0 | -0.001 |
| HDPE | PREWASHED | 0.4% HNO3 | 0.134 | 0.036 | 0.418 | 0.002 | 0 | 0.017 | 0.025 | 0.739 | 0 | 0.001 | 0 | 0.001 | 0.003 | 0 | 0.005 |
| HDPE | PREWASHED | 1 mL of 8M HNO3 | 0.149 | 0.102 | 0.861 | 0.005 | 0 | 0.023 | 0.015 | 1.985 | 0 | 0 | 0 | 0 | 0.001 | 0 | 0.004 |
| | | | | | | | | | | | | | | | | | |
| PETG | NONE | 0.4% HNO3 | 0.014 | 0.003 | 0.213 | 0.087 | 0.121 | -0.006 | 0.072 | 0.288 | 0.003 | 0.001 | 0.001 | 0 | 0.004 | 0 | 0.001 |
| PETG | NONE | 1 mL of 8M HNO3 | 0.006 | 0.029 | 1.242 | 5.269 | 9.099 | 0.041 | 0.09 | 0.138 | 0.002 | 0.001 | 0 | 0 | 0.015 | 0 | -0.001 |
| PETG | EPA | 0.4% HNO3 | -0.041 | -0.001 | -0.267 | 0 | 0.003 | 0.004 | 0.009 | 0.044 | 0 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| PETG | EPA | 1 mL of 8M HNO3 | -0.034 | 0.002 | 0.295 | 2.116 | 3.377 | 0.008 | 0.003 | 0.127 | 0 | 0.001 | 0 | 0 | 0.008 | 0 | 0 |
| PETG | VIRGINIA | 0.4% HNO3 | -0.048 | -0.004 | -0.391 | -0.001 | 0.001 | -0.011 | 0 | 0.009 | -0.001 | 0 | -0.001 | 0 | 0.001 | 0 | 0 |
| PETG | VIRGINIA | 1 mL of 8M HNO3 | -0.054 | -0.001 | 0.001 | 2.736 | 4.504 | 0.01 | -0.003 | 0.057 | -0.001 | 0 | -0.001 | 0 | 0.01 | 0 | -0.001 |
| | | | | | | | | | | | | | | | | | |
| PP | NONE | 0.4% HNO3 | 0.594 | -0.004 | -0.991 | -0.002 | 0 | -0.008 | 0.04 | 0.028 | 0.002 | 0 | 0 | 0 | 0.001 | 0 | 0.001 |
| PP | NONE | 1 mL of 8M HNO3 | 4.225 | 0.01 | -0.17 | -0.001 | -0.001 | -0.001 | 0.087 | 0.029 | -0.002 | -0.001 | 0.001 | 0 | 0.001 | 0 | 0 |
| PP | EPA | 0.4% HNO3 | 0.067 | 0.021 | 0.072 | -0.002 | 0 | -0.005 | 0.002 | 0.008 | 0.001 | -0.001 | 0 | 0 | 0.001 | 0 | 0.001 |
| PP | EPA | 1 mL of 8M HNO3 | 0.724 | 0.02 | 0.128 | -0.002 | 0 | -0.012 | -0.003 | -0.006 | 0 | 0 | 0 | 0 | 0 | 0 | -0.001 |
| PP | VIRGINIA | 0.4% HNO3 | -0.047 | -0.008 | -0.315 | -0.003 | 0 | -0.012 | 0.004 | 0.026 | 0 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| PP | VIRGINIA | 1 mL of 8M HNO3 | 0.843 | 0.002 | 0.032 | -0.005 | -0.001 | -0.013 | -0.002 | -0.003 | -0.003 | 0 | -0.001 | 0 | 0 | 0 | -0.001 |

|  |  |  | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| PETG | NONE | 0.4% HNO3 | 0.014 | 0.003 | 0.213 | 0.067 | 0.121 | -0.006 | 0.072 | 0.288 | 0.003 | 0.001 | 0.001 | 0 | 0.004 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | 0.006 | 0.029 | 1.242 | 5.269 | 9.099 | 0.041 | 0.09 | 0.138 | 0.002 | 0.001 | 0 | 0 | 0.015 | 0 | -0.001 |
| PP | NONE | 0.4% HNO3 | 0.594 | -0.004 | -0.991 | -0.002 | 0 | -0.006 | 0.04 | 0.028 | 0.002 | 0 | 0 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | 4.225 | 0.01 | -0.17 | -0.001 | -0.001 | -0.001 | 0.087 | 0.029 | -0.002 | -0.001 | 0.001 | 0 | 0.001 | 0 | 0 |
| HDPE | NONE | 0.4% HNO3 | -0.017 | 0.001 | 0.642 | -0.002 | -0.001 | -0.006 | 0.02 | 0.008 | 0.001 | 0 | 0.001 | -0.001 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | 0.281 | 0.004 | 0.611 | -0.002 | 0 | -0.003 | 0.033 | -0.001 | -0.001 | 0 | 0 | 0 | 0 | 0 | 0 |
| FEP | NONE | 0.4% HNO3 | 0.053 | 0.068 | 1.886 | 0.014 | 0.03 | 0.493 | 0.19 | 0.099 | -0.001 | 0.01 | 0 | 0.001 | 0.002 | 0 | 0.01 |
|  |  | 1 mL of 8M HNO3 | 0.177 | 0.218 | 41.702 | 0.162 | 0.069 | 0.507 | 1.615 | 0.787 | 0.005 | 0.164 | 0.001 | 0.004 | 0.003 | 0 | 0.084 |
| HDPE | PREWASHED | 0.4% HNO3 | 0.134 | 0.036 | 0.418 | 0.002 | 0 | 0.017 | 0.025 | 0.730 | 0 | 0.001 | 0 | 0.001 | 0.003 | 0 | 0.005 |
|  |  | 1 mL of 8M HNO3 | 0.149 | 0.102 | 0.861 | 0.005 | 0 | 0.023 | 0.015 | 1.985 | 0 | 0 | 0 | 0 | 0.001 | 0 | 0.004 |
| PETG | EPA | 0.4% HNO3 | -0.041 | -0.001 | -0.267 | 0 | 0.003 | 0.004 | 0.009 | 0.044 | 0 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | -0.034 | 0.002 | 0.295 | 2.116 | 3.377 | 0.008 | 0.003 | 0.127 | 0 | 0.001 | 0 | 0 | 0.008 | 0 | 0 |
| PP | EPA | 0.4% HNO3 | 0.067 | 0.021 | 0.072 | -0.002 | 0 | -0.005 | 0.002 | 0.008 | 0.001 | -0.001 | 0 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | 0.724 | 0.02 | 0.128 | -0.002 | 0 | -0.012 | -0.003 | -0.006 | 0 | 0 | 0 | 0 | 0 | 0 | -0.001 |
| HDPE | EPA | 0.4% HNO3 | 0.057 | 0.016 | 0.292 | -0.003 | 0 | -0.009 | 0.004 | 0.013 | -0.002 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | 0.313 | 0.02 | -0.494 | -0.003 | 0 | -0.013 | -0.005 | -0.004 | 0 | 0 | 0 | 0 | 0 | 0 | -0.001 |
| FEP | EPA | 0.4% HNO3 | -0.011 | 0.044 | 0.522 | 0.008 | 0.018 | 0.127 | 0.034 | 0.156 | -0.001 | 0.004 | -0.001 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | -0.002 | 0.075 | 1.156 | 0.011 | 0.04 | 0.246 | 0.032 | 0.012 | 0 | 0.017 | -0.001 | 0.001 | 0 | 0 | 0.002 |
| PETG | VIRGINIA | 0.4% HNO3 | -0.046 | -0.004 | -0.391 | -0.001 | 0.001 | -0.011 | 0 | 0.009 | -0.001 | 0 | -0.001 | 0 | 0.001 | 0 | 0 |
|  |  | 1 mL of 8M HNO3 | -0.064 | -0.001 | 0.001 | 2.736 | 4.504 | 0.01 | -0.003 | 0.057 | -0.001 | 0 | -0.001 | 0 | 0.01 | 0 | -0.001 |
| PP | VIRGINIA | 0.4% HNO3 | -0.047 | -0.006 | -0.315 | -0.003 | 0 | -0.012 | 0.004 | 0.026 | 0 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | 0.843 | 0.002 | 0.032 | -0.005 | -0.001 | -0.013 | -0.002 | -0.003 | -0.003 | 0 | -0.001 | 0 | 0 | 0 | -0.001 |
| HDPE | VIRGINIA | 0.4% HNO3 | -0.014 | -0.009 | -0.126 | -0.004 | 0 | -0.015 | 0.005 | 0.03 | -0.002 | -0.001 | -0.001 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | 0.557 | 0.011 | 0.565 | -0.004 | -0.001 | -0.012 | -0.005 | -0.004 | 0 | -0.001 | -0.001 | 0 | 0 | 0 | -0.001 |
| FEP | VIRGINIA | 0.4% HNO3 | -0.04 | -0.005 | 0.465 | -0.004 | 0.007 | 0.001 | 0.003 | 0.017 | -0.001 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
|  |  | 1 mL of 8M HNO3 | -0.016 | 0.058 | 1.79 | 0.005 | 0.043 | 0.234 | 0.041 | 0.034 | -0.001 | 0.02 | -0.001 | 0.001 | 0 | 0 | 0.001 |
|  |  |  | Al | Cr | Fe | Mn | Co | Ni | Cu | Zn | As | Mo | Ag | Cd | Sb | Tl | Pb |

|  |  |  | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FEP | NONE | 0.4% HNO3 | 0.053 | 0.088 | 1.886 | 0.014 | 0.03 | 0.493 | 0.19 | 0.099 | -0.001 | 0.01 | 0 | 0.001 | 0.002 | 0 | 0.01 |
| FEP | NONE | 1 mL of 8M HNO3 | 0.177 | 0.218 | 41.702 | 0.162 | 0.069 | 0.507 | 1.615 | 0.767 | 0.005 | 0.164 | 0.001 | 0.004 | 0.003 | 0 | 0.084 |
| FEP | EPA | 0.4% HNO3 | -0.011 | 0.044 | 0.522 | 0.008 | 0.018 | 0.127 | 0.034 | 0.158 | -0.001 | 0.004 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| FEP | EPA | 1 mL of 8M HNO3 | -0.002 | 0.075 | 1.158 | 0.011 | 0.04 | 0.246 | 0.032 | 0.012 | 0 | 0.017 | -0.001 | 0.001 | 0 | 0 | 0.002 |
| FEP | VIRGINIA | 0.4% HNO3 | -0.04 | -0.005 | 0.465 | -0.004 | 0.007 | 0.001 | 0.003 | 0.017 | -0.001 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| FEP | VIRGINIA | 1 mL of 8M HNO3 | -0.018 | 0.058 | 1.79 | 0.005 | 0.043 | 0.234 | 0.041 | 0.034 | -0.001 | 0.02 | -0.001 | 0.001 | 0 | 0 | 0.001 |
| HDPE | NONE | 0.4% HNO3 | -0.017 | 0.001 | 0.642 | -0.002 | -0.001 | -0.008 | 0.02 | 0.008 | 0.001 | 0 | 0.001 | -0.001 | 0.001 | 0 | 0.001 |
| HDPE | NONE | 1 mL of 8M HNO3 | 0.281 | 0.004 | 0.611 | -0.002 | 0 | -0.003 | 0.033 | -0.001 | -0.001 | 0 | 0 | 0 | 0 | 0 | 0 |
| HDPE | EPA | 0.4% HNO3 | 0.057 | 0.018 | 0.292 | -0.003 | 0 | -0.009 | 0.004 | 0.013 | -0.002 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| HDPE | EPA | 1 mL of 8M HNO3 | 0.313 | 0.02 | -0.494 | -0.003 | 0 | -0.013 | -0.005 | -0.004 | 0 | 0 | 0 | 0 | 0 | 0 | -0.001 |
| HDPE | VIRGINIA | 0.4% HNO3 | -0.014 | -0.009 | -0.128 | -0.004 | 0 | -0.015 | 0.005 | 0.03 | -0.002 | -0.001 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| HDPE | VIRGINIA | 1 mL of 8M HNO3 | 0.557 | 0.011 | 0.565 | -0.004 | -0.001 | -0.012 | -0.005 | -0.004 | 0 | -0.001 | -0.001 | 0 | 0 | 0 | -0.001 |
| HDPE | PREWASHED | 0.4% HNO3 | 0.134 | 0.038 | 0.418 | 0.002 | 0 | 0.017 | 0.025 | 0.739 | 0 | 0.001 | 0 | 0.001 | 0.003 | 0 | 0.005 |
| HDPE | PREWASHED | 1 mL of 8M HNO3 | 0.149 | 0.102 | 0.861 | 0.005 | 0 | 0.023 | 0.015 | 1.985 | 0 | 0 | 0 | 0 | 0.001 | 0 | 0.004 |
| PETG | NONE | 0.4% HNO3 | 0.014 | 0.003 | 0.213 | 0.067 | 0.121 | -0.008 | 0.072 | 0.288 | 0.003 | 0.001 | 0.001 | 0 | 0.004 | 0 | 0.001 |
| PETG | NONE | 1 mL of 8M HNO3 | 0.008 | 0.029 | 1.242 | 5.269 | 9.099 | 0.041 | 0.09 | 0.138 | 0.002 | 0.001 | 0 | 0 | 0.015 | 0 | -0.001 |
| PETG | EPA | 0.4% HNO3 | -0.041 | -0.001 | -0.267 | 0 | 0.003 | 0.004 | 0.009 | 0.044 | 0 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| PETG | EPA | 1 mL of 8M HNO3 | -0.034 | 0.002 | 0.295 | 2.116 | 3.377 | 0.008 | 0.003 | 0.127 | 0 | 0.001 | 0 | 0 | 0.008 | 0 | 0 |
| PETG | VIRGINIA | 0.4% HNO3 | -0.048 | -0.004 | -0.391 | -0.001 | 0.001 | -0.011 | 0 | 0.009 | -0.001 | 0 | -0.001 | 0 | 0.001 | 0 | 0 |
| PETG | VIRGINIA | 1 mL of 8M HNO3 | -0.054 | -0.001 | 0.001 | 2.736 | 4.504 | 0.01 | -0.003 | 0.057 | -0.001 | 0 | -0.001 | 0 | 0.01 | 0 | -0.001 |
| PP | NONE | 0.4% HNO3 | 0.594 | -0.004 | -0.991 | -0.002 | 0 | -0.008 | 0.04 | 0.028 | 0.002 | 0 | 0 | 0 | 0.001 | 0 | 0.001 |
| PP | NONE | 1 mL of 8M HNO3 | 4.225 | 0.01 | -0.17 | -0.001 | -0.001 | -0.001 | 0.087 | 0.029 | -0.002 | -0.001 | 0.001 | 0 | 0.001 | 0 | 0 |
| PP | EPA | 0.4% HNO3 | 0.067 | 0.021 | 0.072 | -0.002 | 0 | -0.005 | 0.002 | 0.008 | 0.001 | -0.001 | 0 | 0 | 0.001 | 0 | 0.001 |
| PP | EPA | 1 mL of 8M HNO3 | 0.724 | 0.02 | 0.128 | -0.002 | 0 | -0.012 | -0.003 | -0.008 | 0 | 0 | 0 | 0 | 0 | 0 | -0.001 |
| PP | VIRGINIA | 0.4% HNO3 | -0.047 | -0.008 | -0.315 | -0.003 | 0 | -0.012 | 0.004 | 0.026 | 0 | 0 | -0.001 | 0 | 0.001 | 0 | 0.001 |
| PP | VIRGINIA | 1 mL of 8M HNO3 | 0.843 | 0.002 | 0.032 | -0.005 | -0.001 | -0.013 | -0.002 | -0.003 | -0.003 | 0 | -0.001 | 0 | 0 | 0 | -0.001 |

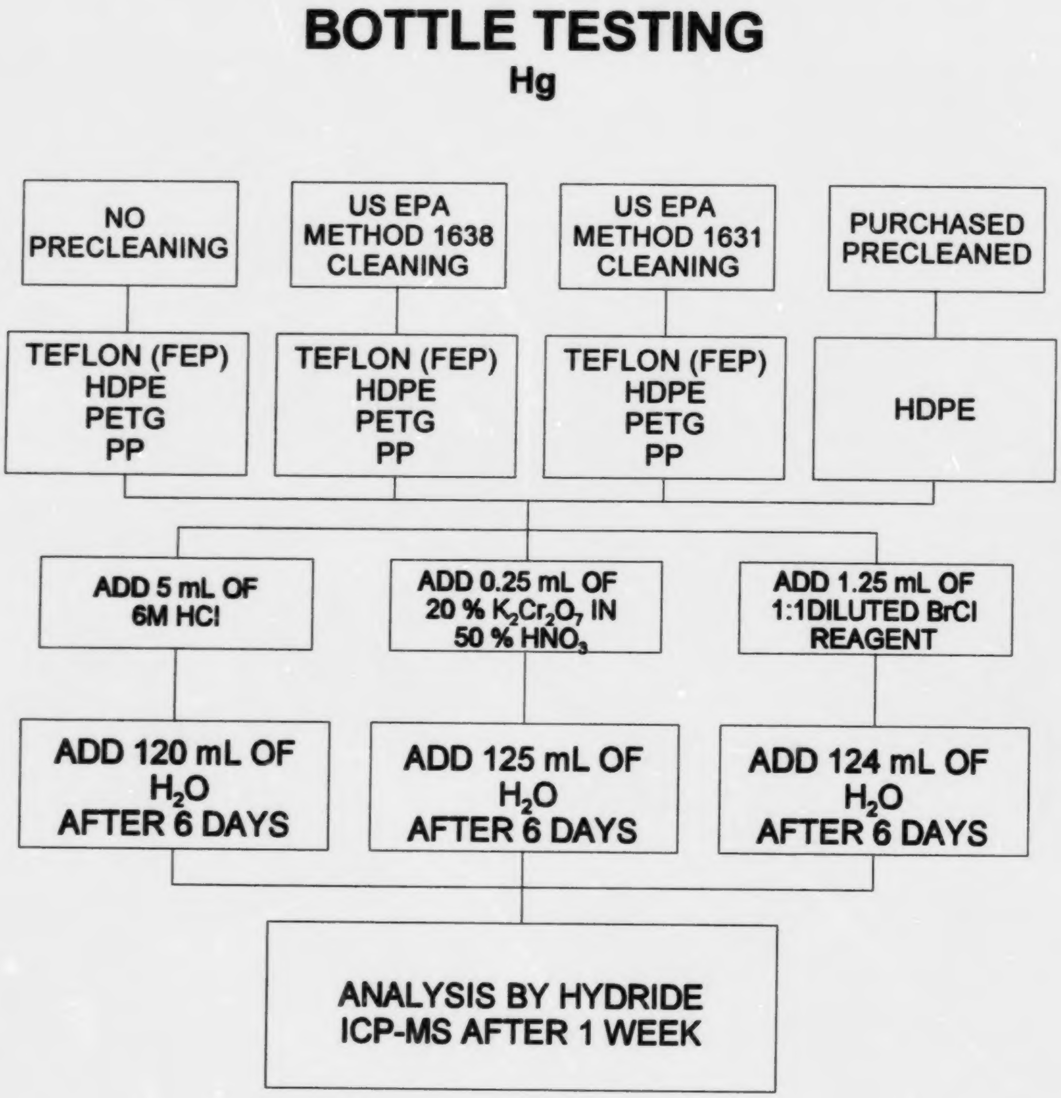
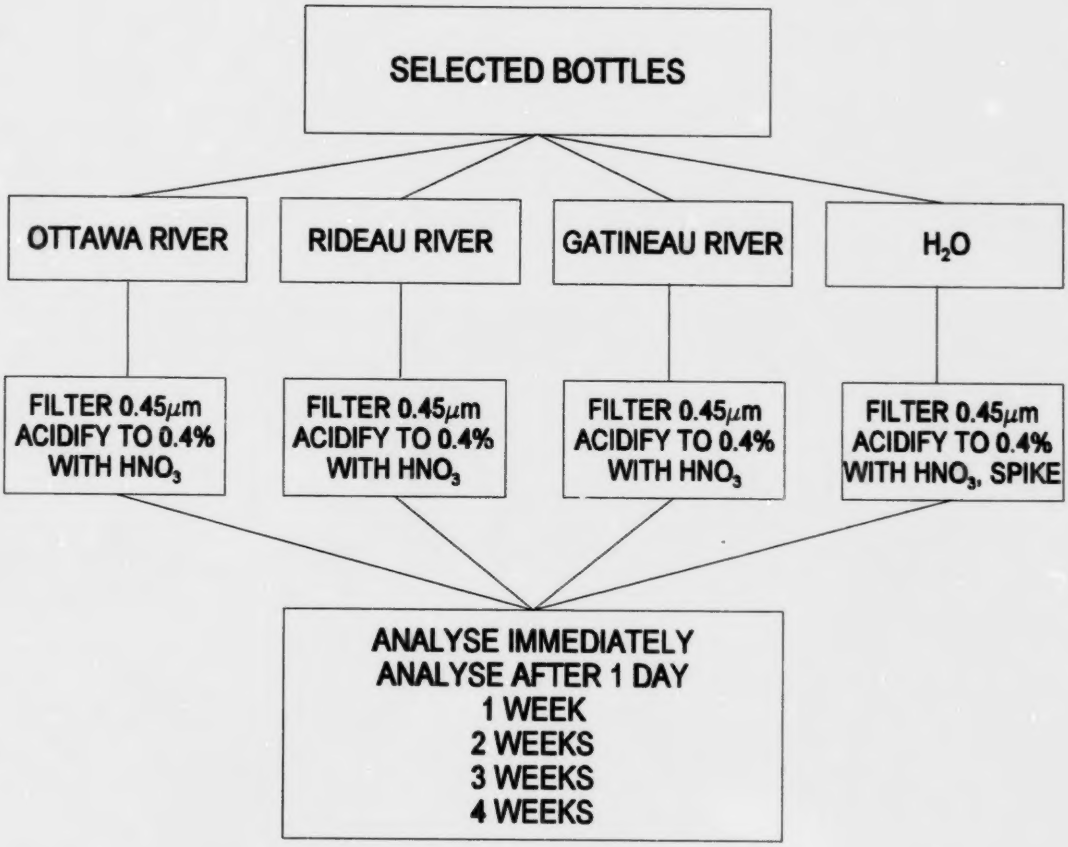

BOTTLE TESTING
Hg
NO PRECLEANING
US EPA METHOD 1638 CLEANING
US EPA METHOD 1631 CLEANING
PURCHASED PRECLEANED
TEFLON (FEP) HDPE PETG PP
TEFLON (FEP) HDPE PETG PP
TEFLON (FEP) HDPE PETG PP
HDPE
ADD 5 mL OF 6M HCl
ADD 0.25 mL OF 20 % $K_2Cr_2O_7$ IN 50 % $HNO_3$
ADD 1.25 mL OF 1:1DILUTED BrCl REAGENT
ADD 120 mL OF $H_2O$ AFTER 6 DAYS
ADD 125 mL OF $H_2O$ AFTER 6 DAYS
ADD 124 mL OF $H_2O$ AFTER 6 DAYS
ANALYSIS BY HYDRIDE ICP-MS AFTER 1 WEEK

Figure 2.4

# PRESERVATION STUDY

## Ag, Al, As, Cd, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Zn, Co, Tl



SELECTED BOTTLES

OTTAWA RIVER

RIDEAU RIVER

GATINEAU RIVER

$H_2O$

FILTER 0.45$\mu$m
ACIDIFY TO 0.4%
WITH $HNO_3$

FILTER 0.45$\mu$m
ACIDIFY TO 0.4%
WITH $HNO_3$

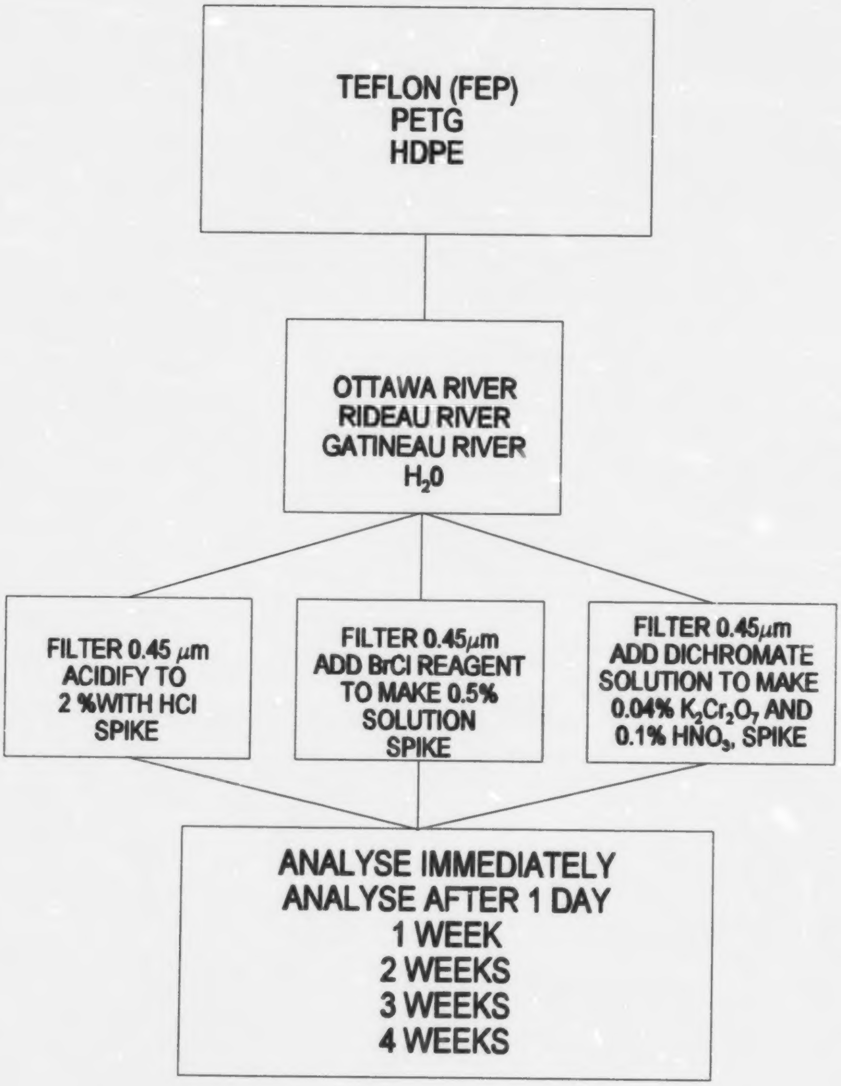
FILTER 0.45$\mu$m
ACIDIFY TO 0.4%
WITH $HNO_3$

FILTER 0.45$\mu$m
ACIDIFY TO 0.4%
WITH $HNO_3$, SPIKE

ANALYSE IMMEDIATELY
ANALYSE AFTER 1 DAY
1 WEEK
2 WEEKS
3 WEEKS
4 WEEKS

# PRESERVATION STUDY

## Hg



TEFLON (FEP)
PETG
HDPE

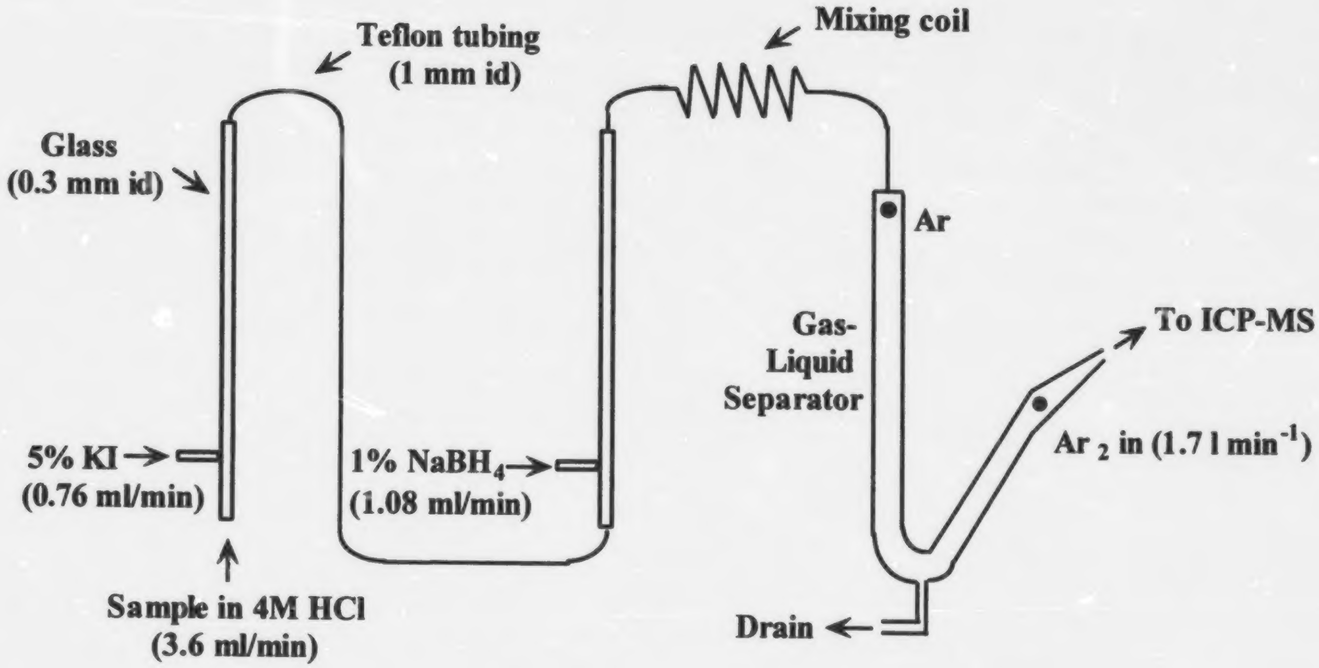
OTTAWA RIVER
RIDEAU RIVER
GATINEAU RIVER
$H_2O$

FILTER 0.45 µm
ACIDIFY TO
2 %WITH HCl
SPIKE

FILTER 0.45µm
ADD BrCl REAGENT
TO MAKE 0.5%
SOLUTION
SPIKE

FILTER 0.45µm
ADD DICHROMATE
SOLUTION TO MAKE
0.04% $K_2Cr_2O_7$ AND
0.1% $HNO_3$, SPIKE

ANALYSE IMMEDIATELY
ANALYSE AFTER 1 DAY
1 WEEK
2 WEEKS
3 WEEKS
4 WEEKS



Teflon tubing
(1 mm id)
Mixing coil
Glass
(0.3 mm id)
Ar
Gas-
Liquid
Separator
To ICP-MS
5% KI
(0.76 ml/min)
1% NaBH$_4$
(1.08 ml/min)
Ar $_2$ in (1.7 l min$^{-1}$)
Sample in 4M HCl
(3.6 ml/min)
Drain

Figure 2.3 Hydride or generation ICP-MS

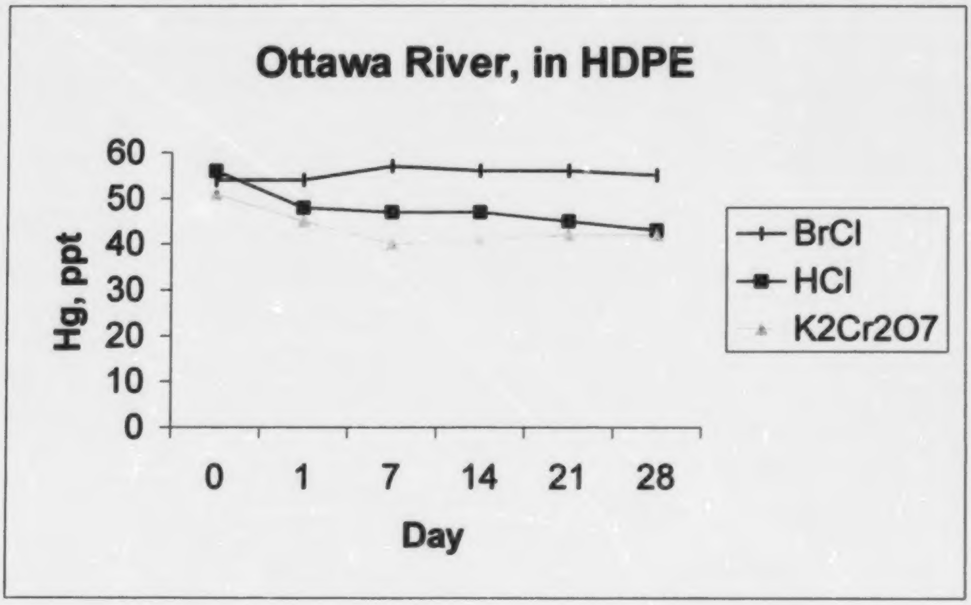


Figure 5.2 - 1

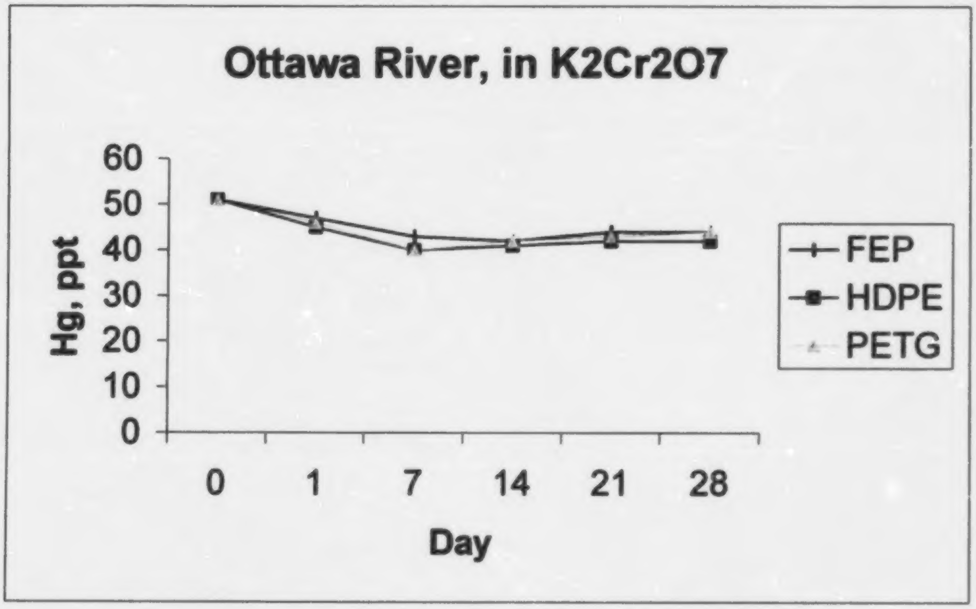


Figure 5.2 - 2

# Bottle contaminants study, Appendix 2.1

All values in ppb, isotopes used indicated

| | | | Al 27 | Cr 52 | Fe 54 | Mn 55 | Fe 56 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| PETG | NONE | 0.4% HNO3 | -0.02911 | 0.007009 | 0.421201 | 0.069939 | 1.662232 | 0.131747 | -0.0089 | 0.064087 | 0.225697 | 0.00144 |
| | | 0.4% HNO3 | 0.033844 | 0.002166 | -0.22272 | 0.057936 | -1.02057 | 0.102277 | -0.00305 | 0.082826 | 0.330574 | 0.003556 |
| | | 0.4% HNO3 | 0.044644 | -0.00084 | 0.306922 | 0.067647 | -0.51112 | 0.119757 | -0.00994 | 0.063175 | 0.507449 | 0.001115 |
| | | 0.4% HNO3 | 0.013008 | 0.004485 | 0.612783 | 0.061677 | 0.043763 | 0.119407 | -0.00487 | 0.080779 | 0.129973 | 0.00403 |
| | | 0.4% HNO3 | 0.009435 | 0.003647 | -0.05372 | 0.078399 | 1.163378 | 0.131981 | -0.00453 | 0.071526 | 0.245608 | 0.005954 |
| | | Mean | 0.014365 | 0.003294 | 0.212894 | 0.06712 | 0.267536 | 0.121034 | -0.00626 | 0.072479 | 0.28786 | 0.003219 |
| | | SD | 0.028338 | 0.002902 | 0.343869 | 0.007897 | 1.125091 | 0.012152 | 0.00299 | 0.009136 | 0.14197 | 0.00199 |
| | | 1 ml 8M | 0.030037 | 0.084719 | 2.368296 | 4.789996 | 1.598682 | 8.205775 | 0.048883 | 0.082191 | 0.153391 | -0.00101 |
| | | 1 ml 8M | -0.00149 | 0.013155 | 0.883803 | 4.940945 | 0.173954 | 8.469541 | 0.0391 | 0.056522 | 0.104048 | 0.004527 |
| | | 1 ml 8M | -0.00092 | 0.018587 | 2.066811 | 5.56872 | 2.000163 | 9.688942 | 0.046026 | 0.116209 | 0.157511 | 0.003635 |
| | | 1 ml 8M | -0.01103 | 0.016844 | 0.602242 | 4.155163 | 1.329965 | 7.127851 | 0.023176 | 0.092084 | 0.123398 | 0.001026 |
| | | 1 ml 8M | 0.012878 | 0.011162 | 0.287956 | 6.892144 | 1.034562 | 12.00421 | 0.049337 | 0.104771 | 0.152189 | 0.001716 |
| | | Mean | 0.005894 | 0.028893 | 1.241822 | 5.269393 | 1.227465 | 9.099263 | 0.041304 | 0.090355 | 0.138107 | 0.001978 |
| | | SD | 0.015963 | 0.031345 | 0.9215 | 1.037078 | 0.687835 | 1.861972 | 0.010929 | 0.022861 | 0.023371 | 0.002189 |
| PP | NONE | 0.4% HNO3 | 0.594599 | 0.007149 | -0.1744 | 8.95E-05 | 0.599189 | 0.000202 | -0.01173 | 0.037254 | 0.045164 | -0.00195 |
| | | 0.4% HNO3 | 0.546991 | -0.00669 | -0.99975 | -0.00111 | -0.9782 | -3.6E-05 | -0.0035 | 0.0336 | 0.022315 | 0.00321 |
| | | 0.4% HNO3 | 0.630278 | -0.00592 | -1.26085 | -0.00251 | -1.20152 | -0.00017 | -0.01032 | 0.046629 | 0.033117 | 0.002516 |
| | | 0.4% HNO3 | 0.637985 | -0.00622 | -1.26903 | -0.00204 | -0.77225 | 1.19E-05 | -0.00044 | 0.022129 | 0.017961 | 0.004676 |
| | | 0.4% HNO3 | 0.561502 | -0.00671 | -1.25128 | -0.0023 | -1.46126 | -0.00057 | -0.00457 | 0.058105 | 0.023458 | 0.00273 |
| | | Mean | 0.594271 | -0.00368 | -0.99106 | -0.00157 | -0.76281 | -0.00011 | -0.00611 | 0.039543 | 0.028403 | 0.002236 |
| | | SD | 0.040363 | 0.006062 | 0.470315 | 0.001072 | 0.803402 | 0.000288 | 0.00476 | 0.013588 | 0.010882 | 0.002488 |
| | | 1 ml 8M | 3.967752 | 0.002381 | -0.88784 | -0.00179 | -1.12924 | -4.7E-05 | -0.0013 | 0.027466 | 0.073802 | -0.00023 |
| | | 1 ml 8M | 4.271387 | 0.015017 | -0.20939 | -0.00153 | 0.724764 | -0.00063 | 0.006723 | 0.003248 | 0.005239 | -0.00317 |
| | | 1 ml 8M | 4.475685 | 0.008683 | -0.27334 | -0.00167 | -0.47645 | -0.00079 | -0.00124 | 0.013507 | 0.001776 | 0.001467 |
| | | 1 ml 8M | 4.258122 | 0.008331 | 0.131116 | 0.000515 | -0.30354 | -0.00044 | -0.00513 | 0.218445 | 0.057201 | -0.0027 |
| | | 1 ml 8M | 4.153874 | 0.014116 | 0.387436 | -0.00165 | 0.649831 | -0.00093 | -0.00156 | 0.170114 | 0.006859 | -0.00326 |
| | | Mean | 4.225364 | 0.009706 | -0.1704 | -0.00122 | -0.10693 | -0.00057 | -0.0005 | 0.086556 | 0.028975 | -0.00158 |

| | | Al 27 | Cr 52 | Fe 54 | Mn 55 | Fe 56 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | SD | 0.185288 | 0.005105 | 0.481905 | 0.000977 | 0.788149 | 0.000344 | 0.004358 | 0.100181 | 0.033906 | 0.002104 |
| HDPE NONE | 0.4% HNO3 | -0.03093 | 0.000675 | 0.241014 | -0.00325 | -0.14776 | -0.00119 | -0.00162 | 0.022265 | 0.0111 | -0.00067 |
| | 0.4% HNO3 | -0.02903 | 0.002487 | 1.15959 | -0.00126 | 0.997012 | -0.00068 | -0.00704 | 0.027046 | 0.012888 | 0.002233 |
| | 0.4% HNO3 | 0.003531 | 0.003421 | 0.604465 | -0.00127 | 0.390561 | -0.00051 | -0.0053 | 0.027599 | 0.0074 | -0.00134 |
| | 0.4% HNO3 | -0.02335 | 0.003968 | 1.436993 | -0.00119 | 0.564533 | 5.94E-05 | -0.00891 | 0.013239 | 0.001959 | 0.003256 |
| | 0.4% HNO3 | -0.00581 | -0.00641 | -0.2345 | -0.00208 | -0.58983 | -0.00038 | -0.00622 | 0.012007 | 0.004604 | 0.001013 |
| | Mean | -0.01712 | 0.000829 | 0.641513 | -0.00181 | 0.242902 | -0.00054 | -0.00582 | 0.020431 | 0.00759 | 0.000898 |
| | SD | 0.015213 | 0.004235 | 0.676453 | 0.000883 | 0.620483 | 0.000454 | 0.002701 | 0.007436 | 0.004502 | 0.001927 |
| | 1 ml 8M | 0.287181 | 0.001076 | 0.053055 | -0.00185 | -0.12903 | -6E-05 | -0.00345 | 0.017413 | -0.00401 | -0.00011 |
| | 1 ml 8M | 0.29665 | 0.006454 | 0.881513 | -0.00081 | 0.765971 | -0.00013 | -0.00154 | 0.056485 | -0.00752 | 0.000226 |
| | 1 ml 8M | 0.294423 | 0.001409 | 1.826709 | -0.00136 | 0.656537 | -0.00022 | -0.00476 | 0.013303 | -0.00522 | -0.00011 |
| | 1 ml 8M | 0.248406 | 0.011206 | 0.8747 | -0.00216 | 1.498956 | 1.22E-05 | -0.0085 | 0.059654 | 0.017391 | -0.00046 |
| | 1 ml 8M | 0.279875 | 0.000382 | -0.58088 | -0.00225 | 0.457331 | -0.00018 | 0.001004 | 0.015932 | -0.00386 | -0.00285 |
| | Mean | 0.281307 | 0.004106 | 0.611019 | -0.00169 | 0.649954 | -0.00012 | -0.00345 | 0.032557 | -0.00064 | -0.00066 |
| | SD | 0.019535 | 0.004643 | 0.915498 | 0.000603 | 0.586988 | 9.31E-05 | 0.00356 | 0.023362 | 0.010188 | 0.001249 |
| FEP NONE | 0.4% HNO3 | 0.14687 | 0.045327 | 0.648838 | 0.009161 | -0.06263 | 0.031543 | 0.20294 | 0.120487 | 0.049294 | 0.00103 |
| | 0.4% HNO3 | 0.013119 | 0.084567 | 1.528219 | 0.02041 | 0.959255 | 0.05137 | 0.189767 | 0.084432 | 0.09716 | -0.0039 |
| | 0.4% HNO3 | -0.00911 | 0.042718 | 1.488656 | 0.011673 | 2.021156 | 0.026431 | 0.595036 | 0.090343 | 0.036414 | 0.000806 |
| | 0.4% HNO3 | 0.085844 | 0.049633 | 2.281611 | 0.011908 | 1.965287 | 0.020711 | 0.146158 | 0.546271 | 0.275804 | -0.00115 |
| | 0.4% HNO3 | 0.028067 | 0.117566 | 3.482942 | 0.015814 | 3.101951 | 0.022323 | 1.328874 | 0.110375 | 0.034096 | -0.00011 |
| | Mean | 0.052959 | 0.067962 | 1.886053 | 0.013793 | 1.597005 | 0.030476 | 0.492555 | 0.190382 | 0.098553 | -0.00067 |
| | SD | 0.063166 | 0.032488 | 1.06339 | 0.004398 | 1.198071 | 0.012409 | 0.501368 | 0.199485 | 0.102301 | 0.002004 |
| | 1 ml 8M | 0.315288 | 0.296966 | 140.2836 | 0.475799 | 161.0209 | 0.075479 | 0.530221 | 1.876824 | 1.211907 | 0.017065 |
| | 1 ml 8M | 0.105865 | 0.13954 | 2.252614 | 0.040349 | 2.682069 | 0.067093 | 0.474139 | 0.073663 | 0.059198 | 0.0008 |
| | 1 ml 8M | 0.083619 | 0.182419 | 3.215954 | 0.019801 | 2.425738 | 0.074394 | 0.559785 | 0.21074 | 0.09711 | 0.002966 |
| | 1 ml 8M | 0.268866 | 0.379428 | 59.95644 | 0.252639 | 60.92397 | 0.077186 | 0.543161 | 0.876615 | 0.506406 | 0.004896 |
| | 1 ml 8M | 0.109042 | 0.093815 | 2.800491 | 0.02022 | 3.421863 | 0.052453 | 0.428726 | 5.035339 | 1.95885 | -0.00182 |
| | Mean | 0.176536 | 0.218434 | 41.70182 | 0.161762 | 46.0949 | 0.069321 | 0.507206 | 1.614636 | 0.766694 | 0.004782 |

| | | Al 27 | Cr 52 | Fe 54 | Mn 55 | Fe 56 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | SD | 0.107192 | 0.117407 | 60.42002 | 0.201126 | 68.99373 | 0.010186 | 0.054405 | 2.040842 | 0.811803 | 0.007308 |
| HDPE PREWASH | 0.4% HNO3 | 0.123098 | 0.04631 | -0.57199 | -0.00021 | -0.07385 | -6.2E-05 | 0.014785 | 0.030544 | 0.836784 | 0.000227 |
| | 0.4% HNO3 | 0.217027 | 0.036698 | 0.378319 | 0.005708 | 1.016738 | 2.5E-05 | 0.017425 | 0.028286 | 0.637337 | 0.000906 |
| | 0.4% HNO3 | 0.12386 | 0.031192 | 0.6948 | 0.001273 | -0.48223 | -3.7E-05 | 0.015102 | 0.020466 | 0.466897 | 0.001243 |
| | 0.4% HNO3 | 0.110111 | 0.032319 | 0.727853 | 0.002711 | 0.184511 | 0.000112 | 0.022468 | 0.02704 | 0.978984 | -0.0018 |
| | 0.4% HNO3 | 0.094376 | 0.033137 | 0.858636 | 0.001087 | 0.06488 | -0.00057 | 0.016633 | 0.016857 | 0.77698 | 0.000562 |
| | Mean | 0.133694 | 0.035931 | 0.417524 | 0.002114 | 0.14201 | -0.00011 | 0.017283 | 0.024639 | 0.739397 | 0.000227 |
| | SD | 0.048103 | 0.006157 | 0.580611 | 0.00226 | 0.549814 | 0.000269 | 0.003095 | 0.005743 | 0.195557 | 0.001197 |
| | 1 ml 8M | 0.099997 | 0.099379 | 1.894445 | 0.003441 | 0.602349 | 4.98E-05 | 0.027937 | 0.005445 | 1.112387 | 0.000337 |
| | 1 ml 8M | 0.149301 | 0.131782 | 0.192141 | 0.003433 | 0.609697 | 0.000473 | 0.02079 | 0.023698 | 3.894879 | 3.61E-09 |
| | 1 ml 8M | 0.129631 | 0.07999 | 0.239995 | 0.002585 | 0.011582 | -0.00053 | 0.009557 | 0.012196 | 1.234597 | 0.00157 |
| | 1 ml 8M | 0.15083 | 0.121367 | 1.011897 | 0.00133 | 0.69531 | -2.5E-05 | 0.019699 | 0.009039 | 2.354383 | -0.00381 |
| | 1 ml 8M | 0.213738 | 0.079892 | 0.967734 | 0.01565 | 0.485134 | -0.00035 | 0.035981 | 0.022733 | 1.328764 | 0.002576 |
| | Mean | 0.148699 | 0.102482 | 0.861242 | 0.005288 | 0.480814 | -7.7E-05 | 0.022793 | 0.014622 | 1.985002 | 0.000134 |
| | SD | 0.041745 | 0.02367 | 0.695555 | 0.005857 | 0.272758 | 0.000389 | 0.009864 | 0.008207 | 1.176784 | 0.002431 |
| PETG EPA | 0.4% HNO3 | -0.02774 | -0.0058 | -0.85015 | -0.00121 | -0.4533 | 0.000657 | 0.009075 | 0.009685 | 0.01944 | -0.00067 |
| | 0.4% HNO3 | -0.04672 | -0.00544 | -0.70043 | 0.000385 | -0.20674 | 0.003208 | 0.008346 | 0.011452 | 0.036549 | -0.00212 |
| | 0.4% HNO3 | -0.04961 | 0.002377 | 0.283146 | -0.00028 | 0.395727 | 0.001906 | -0.00449 | 0.005367 | 0.0395 | -0.00101 |
| | 0.4% HNO3 | -0.02358 | 0.005518 | 0.471497 | 0.001836 | 0.474336 | 0.005008 | 0.00512 | 0.009664 | 0.082084 | 0.001005 |
| | 0.4% HNO3 | -0.05892 | -0.00216 | -0.53973 | 3.13E-05 | 0.066071 | 0.002915 | 0.002164 | 0.007736 | 0.041562 | 0.003906 |
| | Mean | -0.04131 | -0.0011 | -0.26713 | 0.000153 | 0.05522 | 0.002739 | 0.004043 | 0.008781 | 0.043827 | 0.000222 |
| | SD | 0.015055 | 0.004947 | 0.602151 | 0.001111 | 0.393361 | 0.001614 | 0.005508 | 0.002317 | 0.023104 | 0.002345 |
| | 1 ml 8M | -0.03653 | 0.008872 | 1.186852 | 3.222497 | 1.18517 | 5.215042 | 0.012289 | 0.004341 | 0.521388 | 0.004225 |
| | 1 ml 8M | -0.03624 | -0.00145 | -0.02392 | 2.095352 | 0.030769 | 3.326199 | 0.008582 | 0.00217 | 0.01551 | -0.00067 |
| | 1 ml 8M | -0.02865 | 0.002387 | -0.47062 | 1.632454 | 0.239713 | 2.604309 | 0.012889 | 0.004085 | 0.03786 | -0.002 |
| | 1 ml 8M | -0.04272 | 0.001124 | -0.05058 | 1.686276 | 0.553996 | 2.645223 | 0.00017 | 0.003479 | 0.031571 | -0.00077 |
| | 1 ml 8M | -0.02475 | 0.001487 | 0.830942 | 1.942004 | 1.10014 | 3.094447 | 0.006574 | 0.00131 | 0.026306 | -0.00066 |
| | Mean | -0.03378 | 0.002483 | 0.294537 | 2.115717 | 0.621958 | 3.377044 | 0.008101 | 0.003077 | 0.126527 | 2.51E-05 |

| | | | Al 27 | Cr 52 | Fe 54 | Mn 55 | Fe 56 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SD | 0.007096 | 0.003846 | 0.687374 | 0.646822 | 0.511395 | 1.071607 | 0.005146 | 0.001296 | 0.220886 | 0.002414 |
| PP | EPA | 0.4% HNO3 | 0.043186 | 0.019259 | 0.601745 | -0.00213 | 2.131618 | 0.000124 | -0.01483 | 0.003425 | 0.008199 | 0.001103 |
| | | 0.4% HNO3 | 0.080914 | 0.020292 | -0.83431 | -0.00252 | -0.5652 | -0.00023 | -0.00141 | 0.002215 | 0.005828 | 0.004297 |
| | | 0.4% HNO3 | 0.066821 | 0.020448 | -0.37184 | -0.00226 | 0.874051 | -0.00072 | -0.00435 | 0.002566 | 0.00439 | -0.00121 |
| | | 0.4% HNO3 | 0.040377 | 0.020509 | 0.417324 | -0.00125 | 0.777883 | -0.00045 | 0.001073 | -0.00171 | 0.008183 | -0.00121 |
| | | 0.4% HNO3 | 0.102917 | 0.02364 | 0.548781 | -0.00274 | 1.208827 | -0.00038 | -0.0044 | 0.005983 | 0.011718 | -0.00044 |
| | | Mean | 0.066843 | 0.02083 | 0.072341 | -0.00218 | 0.885435 | -0.00033 | -0.00478 | 0.002496 | 0.007664 | 0.000509 |
| | | SD | 0.026265 | 0.001651 | 0.641401 | 0.00057 | 0.971288 | 0.000308 | 0.006062 | 0.002775 | 0.002788 | 0.002319 |
| | | 1 ml 8M | 0.696017 | 0.022598 | -0.40356 | -0.00239 | 1.134731 | 0.000161 | -0.01747 | -0.00407 | -0.00783 | -0.00307 |
| | | 1 ml 8M | 0.715839 | 0.02389 | 0.595712 | -0.00223 | 0.95933 | -9.9E-05 | -0.00814 | -0.00457 | -0.00682 | 0.002191 |
| | | 1 ml 8M | 0.827932 | 0.022046 | 0.33632 | -0.00185 | 1.489528 | -0.00011 | -0.0133 | -0.00468 | -0.00649 | 0.002631 |
| | | 1 ml 8M | 0.662773 | 0.017024 | -0.15648 | -0.00247 | 0.630028 | -0.00014 | -0.01009 | 0.001308 | -0.00025 | 0.000329 |
| | | 1 ml 8M | 0.719643 | 0.015797 | 0.266713 | -0.00233 | -0.23298 | -0.00026 | -0.00885 | -0.00468 | -0.0086 | -0.00242 |
| | | Mean | 0.724441 | 0.020271 | 0.127741 | -0.00225 | 0.796127 | -8.9E-05 | -0.01157 | -0.00334 | -0.006 | -6.6E-05 |
| | | SD | 0.062085 | 0.003613 | 0.401522 | 0.00024 | 0.653595 | 0.000153 | 0.003843 | 0.00261 | 0.00332 | 0.0026 |
| HDPE | EPA | 0.4% HNO3 | 0.084179 | 0.016442 | 1.127958 | -0.00317 | 1.26666 | 7.4E-05 | -0.00643 | 0.002971 | 0.012996 | -0.00132 |
| | | 0.4% HNO3 | 0.020219 | 0.020236 | 0.199492 | -0.00158 | 1.969622 | -0.00049 | -0.01665 | 0.006094 | 0.009962 | -0.00154 |
| | | 0.4% HNO3 | 0.039917 | 0.015721 | -0.23789 | -0.00523 | 0.078185 | -0.00075 | -0.01228 | 0.003577 | 0.009882 | -0.00308 |
| | | 0.4% HNO3 | 0.050466 | 0.015067 | 0.241591 | -0.00297 | 0.610646 | -0.00018 | -0.00998 | 0.004133 | 0.0109 | -0.00176 |
| | | 0.4% HNO3 | 0.089553 | 0.014331 | 0.13078 | -0.00287 | 0.098097 | -0.00049 | 0.000446 | 0.004891 | 0.019949 | -0.00099 |
| | | Mean | 0.056867 | 0.016359 | 0.292386 | -0.00317 | 0.804642 | -0.00037 | -0.00898 | 0.004333 | 0.012738 | -0.00174 |
| | | SD | 0.02952 | 0.002304 | 0.504158 | 0.001316 | 0.811668 | 0.000319 | 0.006445 | 0.001213 | 0.004222 | 0.000803 |
| | | 1 ml 8M | 0.29141 | 0.021026 | -0.19015 | -0.00316 | -0.03428 | -0.0007 | -0.00941 | -0.00368 | 0.012346 | 0.000331 |
| | | 1 ml 8M | 0.282537 | 0.024748 | -0.89242 | -0.00369 | -0.64775 | -0.00039 | -0.01668 | -0.00616 | -0.00567 | -0.00199 |
| | | 1 ml 8M | 0.324866 | 0.022053 | 0.063216 | -0.00286 | -0.495 | -0.00054 | -0.01169 | -0.00348 | -0.00983 | -0.00066 |
| | | 1 ml 8M | 0.333681 | 0.014895 | -0.8629 | -0.00343 | -1.45456 | 0.000111 | -0.01654 | -0.00338 | -0.00763 | 0.002533 |
| | | 1 ml 8M | 0.332111 | 0.018332 | -0.58801 | -0.00298 | -1.7645 | -0.00042 | -0.01137 | -0.0062 | -0.00721 | -0.00033 |
| | | Mean | 0.312921 | 0.020211 | -0.49405 | -0.00323 | -0.87922 | -0.00039 | -0.01314 | -0.00458 | -0.0036 | -2.3E-05 |

| | | | Al 27 | Cr 52 | Fe 54 | Mn 55 | Fe 56 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SD | 0.024124 | 0.003756 | 0.420262 | 0.000335 | 0.712367 | 0.000305 | 0.00329 | 0.001463 | 0.009036 | 0.00166 |
| FEP | EPA | 0.4% HNO3 | -0.00192 | 0.062872 | 1.331688 | 0.003501 | 0.475201 | 0.012054 | 0.131942 | 0.011688 | 0.005347 | -0.00099 |
| | | 0.4% HNO3 | -0.01432 | 0.076771 | 1.220019 | 0.014985 | 1.660373 | 0.023343 | 0.136186 | 0.01692 | 0.012567 | 0.003405 |
| | | 0.4% HNO3 | -0.0199 | 0.020445 | -0.39539 | 0.005688 | -0.07684 | 0.017573 | 0.160501 | 0.02089 | 0.008241 | -0.00406 |
| | | 0.4% HNO3 | -0.01286 | 0.03103 | -0.50584 | 0.001836 | -0.61098 | 0.015522 | 0.105543 | 0.067626 | 0.747681 | -0.0034 |
| | | 0.4% HNO3 | -0.00452 | 0.028995 | 0.960988 | 0.012991 | 0.163362 | 0.019722 | 0.100147 | 0.054622 | 0.006805 | -0.00241 |
| | | Mean | -0.0107 | 0.044023 | 0.522293 | 0.0078 | 0.322224 | 0.017643 | 0.126864 | 0.034349 | 0.156128 | -0.00149 |
| | | SD | 0.007374 | 0.024384 | 0.89911 | 0.005854 | 0.846966 | 0.004259 | 0.024559 | 0.025084 | 0.330699 | 0.002971 |
| | | 1 ml 8M | -0.02545 | 0.089174 | 2.569279 | 0.016891 | 1.554778 | 0.061144 | 0.291525 | 0.017747 | 0.017021 | -0.00022 |
| | | 1 ml 8M | 0.033701 | 0.092129 | 0.23454 | 0.007509 | 0.894876 | 0.036948 | 0.278426 | 0.020554 | 0.021799 | 0.000437 |
| | | 1 ml 8M | -0.0149 | 0.058857 | 2.826684 | 0.014948 | 3.111307 | 0.033938 | 0.214453 | 0.025067 | 0.013547 | 0.00153 |
| | | 1 ml 8M | -0.00926 | 0.077321 | -0.08915 | 0.004715 | 0.253283 | 0.040507 | 0.201202 | 0.031442 | 0.002127 | 0.00109 |
| | | 1 ml 8M | 0.00464 | 0.057075 | 0.237906 | 0.008691 | 0.06327 | 0.029384 | 0.243476 | 0.063644 | 0.007481 | -0.00174 |
| | | Mean | -0.00225 | 0.074911 | 1.155852 | 0.010551 | 1.175503 | 0.040384 | 0.245816 | 0.031691 | 0.012395 | 0.000219 |
| | | SD | 0.022844 | 0.016443 | 1.416944 | 0.005155 | 1.230599 | 0.012302 | 0.039154 | 0.018597 | 0.007758 | 0.001281 |
| PETG | VIRGINIA | 0.4% HNO3 | -0.04909 | -0.00822 | -0.2185 | 0.000145 | -1.01891 | 0.000308 | -0.01361 | -0.0006 | 0.004841 | -0.00218 |
| | | 0.4% HNO3 | -0.04313 | -0.00078 | 0.353722 | -0.00066 | -0.46287 | 0.000617 | -0.01541 | 0.004885 | 0.009504 | 0.003048 |
| | | 0.4% HNO3 | -0.04712 | -0.00037 | -0.37751 | 0.000364 | -0.2596 | 0.004746 | -0.00804 | -0.00229 | 0.017804 | -0.00392 |
| | | 0.4% HNO3 | -0.04712 | -0.00673 | -1.10524 | -0.00368 | -1.26567 | -0.00058 | -0.00636 | 4.97E-05 | 0.005336 | 0.001196 |
| | | 0.4% HNO3 | -0.05338 | -0.00517 | -0.6075 | -0.00279 | -0.66497 | 0.002057 | -0.01278 | -0.00342 | 0.007701 | -0.00228 |
| | | Mean | -0.04797 | -0.00425 | -0.39101 | -0.00133 | -0.7344 | 0.00143 | -0.01124 | -0.00028 | 0.009037 | -0.00083 |
| | | SD | 0.003723 | 0.003528 | 0.53422 | 0.001813 | 0.408405 | 0.002082 | 0.003853 | 0.003195 | 0.005248 | 0.002855 |
| | | 1 ml 8M | -0.05585 | -0.00409 | 0.102087 | 1.768658 | -0.43112 | 2.829762 | 0.001126 | -0.00411 | 0.040498 | -0.00109 |
| | | 1 ml 8M | -0.05852 | -0.0015 | 0.498811 | 2.712578 | 0.167068 | 4.389307 | 0.009348 | -0.00173 | 0.011995 | 0.002605 |
| | | 1 ml 8M | -0.05654 | 0.001002 | -0.49309 | 2.378105 | -0.24328 | 3.856454 | 0.007041 | -0.00514 | 0.216812 | -0.00184 |
| | | 1 ml 8M | -0.03804 | 0.002311 | 0.08287 | 4.339392 | -1.43386 | 7.396494 | 0.024679 | -0.00242 | 0.008853 | -0.00488 |
| | | 1 ml 8M | -0.06292 | -0.0045 | -0.18698 | 2.479786 | -0.46946 | 4.048092 | 0.009246 | -0.00292 | 0.008356 | 0.00184 |
| | | Mean | -0.05438 | -0.00135 | 0.000739 | 2.735704 | -0.48213 | 4.504022 | 0.010288 | -0.00326 | 0.057303 | -0.00067 |

|  |  |  | Al 27 | Cr 52 | Fe 54 | Mn 55 | Fe 56 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  | SD | 0.009538 | 0.003016 | 0.368849 | 0.961886 | 0.588866 | 1.718278 | 0.008711 | 0.001362 | 0.090169 | 0.003013 |
| PP | VIRGINIA | 0.4% HNO3 | -0.08245 | -0.00843 | -0.4061 | -0.0036 | -1.32068 | -0.00043 | -0.01962 | -0.0046 | 0.00414 | 0.000757 |
|  |  | 0.4% HNO3 | -0.03335 | -0.00696 | 0.451544 | -0.00163 | -1.06261 | 0.000419 | -0.0097 | 0.007041 | 0.032556 | -0.00184 |
|  |  | 0.4% HNO3 | -0.06104 | -0.00505 | -0.04589 | -0.00412 | -0.14953 | -0.00047 | -0.01472 | 0.00581 | 0.03081 | 0.002377 |
|  |  | 0.4% HNO3 | -0.0337 | -0.00995 | -0.94869 | -0.00455 | -0.66646 | -0.00056 | -0.00705 | 0.005869 | 0.028721 | -0.0013 |
|  |  | 0.4% HNO3 | -0.02525 | -0.00867 | -0.62424 | -0.00338 | -1.45827 | -0.00058 | -0.01106 | 0.00528 | 0.033498 | 0.000863 |
|  |  | Mean | -0.04716 | -0.00781 | -0.31468 | -0.00346 | -0.93151 | -0.00032 | -0.01243 | 0.003879 | 0.025945 | 0.000173 |
|  |  | SD | 0.023925 | 0.001874 | 0.539794 | 0.001115 | 0.531004 | 0.00042 | 0.004879 | 0.004786 | 0.012325 | 0.001723 |
|  |  | 1 ml 8M | 0.991007 | 0.005651 | 0.579632 | -0.00398 | -0.53491 | -0.00046 | -0.01332 | -0.00294 | -0.00382 | -0.00173 |
|  |  | 1 ml 8M | 0.732723 | 0.006146 | 0.107786 | -0.00513 | -0.9005 | -0.00062 | -0.01236 | 0.011443 | -0.00799 | -0.00367 |
|  |  | 1 ml 8M | 0.870237 | 0.002445 | -0.66625 | -0.0054 | -1.18965 | -0.00046 | -0.01569 | -0.00736 | 0.005358 | -0.00453 |
|  |  | 1 ml 8M | 0.768372 | -0.002 | -0.25317 | -0.00511 | -1.62891 | -0.00069 | -0.01445 | -0.00722 | -0.00485 | -0.00291 |
|  |  | 1 ml 8M | 0.851998 | 3.97E-08 | 0.392852 | -0.00455 | -1.00707 | -0.00068 | -0.00909 | -0.00577 | -0.00349 | 4.62E-09 |
|  |  | Mean | 0.842867 | 0.002448 | 0.032171 | -0.00483 | -1.05221 | -0.00058 | -0.01298 | -0.00237 | -0.00296 | -0.00256 |
|  |  | SD | 0.100572 | 0.003527 | 0.501181 | 0.000569 | 0.401289 | 0.000117 | 0.002507 | 0.007923 | 0.004977 | 0.001765 |
| HDPE | VIRGINIA | 0.4% HNO3 | -0.00439 | -0.01106 | -0.15751 | -0.00379 | -1.25222 | -0.00042 | -0.00792 | 0.004222 | 0.033472 | 0.000863 |
|  |  | 0.4% HNO3 | -0.00697 | -0.00995 | -0.04885 | -0.00431 | -0.91248 | -0.00036 | -0.01211 | 0.006382 | 0.032891 | -0.00367 |
|  |  | 0.4% HNO3 | -0.01518 | -0.00426 | 0.442713 | -0.00319 | -0.81438 | -0.00061 | -0.02011 | 0.009848 | 0.042482 | -0.00422 |
|  |  | 0.4% HNO3 | -0.01972 | -0.01093 | -0.58561 | -0.0033 | -1.59118 | -0.00036 | -0.01717 | 0.001758 | 0.018216 | -0.00098 |
|  |  | 0.4% HNO3 | -0.02185 | -0.01022 | -0.28103 | -0.00399 | -1.3587 | -0.00051 | -0.01878 | 0.00226 | 0.025328 | -0.00109 |
|  |  | Mean | -0.01362 | -0.00928 | -0.12606 | -0.00372 | -1.18579 | -0.00045 | -0.01522 | 0.004894 | 0.030478 | -0.00182 |
|  |  | SD | 0.007694 | 0.002847 | 0.376051 | 0.000472 | 0.320662 | 0.000106 | 0.005083 | 0.003316 | 0.009162 | 0.002101 |
|  |  | 1 ml 8M | 1.939142 | 0.040882 | 0.815049 | -0.00299 | -0.56672 | -0.00059 | 0.019983 | -0.00573 | 0.00274 | 0.00087 |
|  |  | 1 ml 8M | 0.218511 | 0.00724 | 0.875642 | -0.00489 | 0.377623 | -0.00067 | -0.0217 | -0.00738 | -0.00754 | -0.00087 |
|  |  | 1 ml 8M | 0.193973 | 0.002479 | -0.30946 | -0.00506 | 0.342207 | -0.00077 | -0.02007 | 0.002711 | -0.006 | -0.00196 |
|  |  | 1 ml 8M | 0.255542 | 0.004491 | 0.40453 | -0.00449 | -0.3322 | -0.00078 | -0.01616 | -0.00768 | -0.00575 | 0.001093 |
|  |  | 1 ml 8M | 0.178733 | 0.002292 | 1.037548 | -0.00419 | -0.05222 | -0.0008 | -0.02096 | -0.00672 | -0.00592 | -0.00109 |
|  |  | Mean | 0.55718 | 0.011477 | 0.564663 | -0.00432 | -0.04626 | -0.00072 | -0.01178 | -0.00496 | -0.00449 | -0.00039 |

| | | | Al 27 | Cr 52 | Fe 54 | Mn 55 | Fe 56 | Co 59 | Ni 60 | Cu 65 | Zn 66 | As 75 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SD | 0.773085 | 0.016558 | 0.541468 | 0.000818 | 0.413297 | 8.78E-05 | 0.017885 | 0.004353 | 0.004107 | 0.001322 |
| FEP | VIRGINIA | 0.4% HNO3 | -0.0343 | -0.0077 | 0.85756 | -0.00535 | 0.446388 | 0.000555 | -0.00507 | 0.007276 | 0.02189 | 0.000986 |
| | | 0.4% HNO3 | -0.02955 | -0.00527 | 0.59858 | -0.00363 | 0.358203 | 0.002279 | 0.009588 | 0.00372 | 0.01661 | -0.00033 |
| | | 0.4% HNO3 | -0.03006 | -0.005 | 0.603818 | -0.00466 | 0.188863 | 0.008663 | 0.008849 | 0.004781 | 0.016585 | 0.000218 |
| | | 0.4% HNO3 | -0.05823 | 0.00243 | 0.077203 | -0.00277 | 0.946388 | 0.01357 | -0.00411 | -0.00126 | 0.013059 | -0.00022 |
| | | 0.4% HNO3 | -0.04708 | -0.01085 | 0.190021 | -0.00386 | 0.364866 | 0.009701 | -0.00548 | 0.002623 | 0.014999 | -0.00479 |
| | | Mean | -0.03984 | -0.00528 | 0.465436 | -0.00405 | 0.460941 | 0.006954 | 0.000754 | 0.003428 | 0.016629 | -0.00083 |
| | | SD | 0.012478 | 0.004908 | 0.322961 | 0.000991 | 0.28709 | 0.005409 | 0.007748 | 0.003136 | 0.003282 | 0.002275 |
| | | 1 ml 8M | -0.00453 | 0.060137 | 2.128826 | 0.003201 | 1.066903 | 0.057592 | 0.288576 | 0.029944 | 0.023825 | -0.0013 |
| | | 1 ml 8M | -0.03592 | 0.052912 | 2.922029 | 0.009595 | 3.185712 | 0.048106 | 0.229782 | 0.034981 | 0.060405 | -0.00228 |
| | | 1 ml 8M | 0.029145 | 0.055863 | 0.605931 | 0.004117 | 1.045628 | 0.040233 | 0.224646 | 0.082387 | 0.012469 | -0.00065 |
| | | 1 ml 8M | -0.01775 | 0.078801 | 1.522551 | 0.006406 | 0.764635 | 0.043484 | 0.258058 | 0.036679 | 0.075041 | 0.000973 |
| | | 1 ml 8M | -0.05194 | 0.042975 | 1.772561 | 0.001342 | 0.247088 | 0.025527 | 0.166822 | 0.018765 | -0.00076 | -0.00324 |
| | | Mean | -0.0162 | 0.058138 | 1.790379 | 0.004932 | 1.261993 | 0.042988 | 0.233577 | 0.040551 | 0.034196 | -0.0013 |
| | | SD | 0.031069 | 0.013166 | 0.847075 | 0.003181 | 1.125052 | 0.01175 | 0.045207 | 0.02441 | 0.032237 | 0.001605 |

All values in ppb, isotopes used

| | | | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|
| PETG | NONE | 0.4% HNO3 | 0.003066 | 0.001612 | 0.000638 | 0.005791 | 0.000161 | 0.001137 |
| | | 0.4% HNO3 | 0.001272 | 0.00228 | -0.00035 | 0.004433 | 0.000362 | 0.001654 |
| | | 0.4% HNO3 | 0.001273 | 0.000755 | 0.00085 | 0.00374 | 0.000332 | 0.000834 |
| | | 0.4% HNO3 | 0.000487 | 0.00065 | 0.000496 | 0.002785 | 0.000141 | 0.000603 |
| | | 0.4% HNO3 | -0.00053 | 0.00044 | 0.000248 | 0.003106 | 0.0001 | 0.001106 |
| | | Mean | 0.001115 | 0.001148 | 0.000375 | 0.003971 | 0.000219 | 0.001067 |
| | | SD | 0.001317 | 0.000775 | 0.000463 | 0.001198 | 0.000119 | 0.000394 |
| | | 1 ml 8M | 0.005593 | 0.000863 | -0.00039 | 0.014462 | 0.00013 | -0.00067 |
| | | 1 ml 8M | 0.000301 | -0.00026 | -0.00035 | 0.014412 | 0.000441 | -0.0004 |
| | | 1 ml 8M | 0.000113 | 0.00067 | 0.000319 | 0.014828 | 0.00022 | -0.00059 |
| | | 1 ml 8M | 0.000602 | 0.000618 | 0.000354 | 0.012654 | 0.00013 | -0.00064 |
| | | 1 ml 8M | -0.00041 | 0.000177 | 0.000106 | 0.017639 | 0.00023 | -0.0006 |
| | | Mean | 0.001239 | 0.000413 | 7.04E-06 | 0.014799 | 0.00023 | -0.00058 |
| | | SD | 0.002462 | 0.000454 | 0.000359 | 0.001798 | 0.000127 | 0.000107 |
| PP | NONE | 0.4% HNO3 | -0.00079 | 3.03E-10 | -3.5E-05 | 0.001369 | 0.00018 | 0.00053 |
| | | 0.4% HNO3 | 0.00053 | 0.000889 | 0 | 0.002491 | 0.0001 | 0.001353 |
| | | 0.4% HNO3 | 0.000228 | 0.00057 | -0.00018 | 0.001587 | 0.000402 | 0.001574 |
| | | 0.4% HNO3 | 0.000799 | 0.000697 | -0.00025 | 0.000766 | 7.06E-05 | 0.001767 |
| | | 0.4% HNO3 | -0.00053 | -1.8E-05 | -0.00061 | 0.001035 | 0.000111 | 0.001118 |
| | | Mean | 4.64E-05 | 0.000428 | -0.00021 | 0.00145 | 0.000173 | 0.001268 |
| | | SD | 0.000684 | 0.000415 | 0.000242 | 0.000661 | 0.000134 | 0.000479 |
| | | 1 ml 8M | -0.00076 | 0.00113 | -0.00054 | 0.001453 | 0.000264 | -0.00042 |
| | | 1 ml 8M | -0.00042 | 0.00178 | -0.00029 | 0.000416 | -3E-05 | -0.00016 |
| | | 1 ml 8M | -0.00042 | 0.001694 | -0.00029 | 0.000358 | -2E-05 | -0.00082 |
| | | 1 ml 8M | -0.00077 | 0.00065 | 0.000108 | 0.000509 | 0.000143 | 0.002322 |
| | | 1 ml 8M | -0.00058 | 0.000543 | -0.0004 | 0.0003 | 0.000236 | -0.00055 |
| | | Mean | -0.00059 | 0.001159 | -0.00028 | 0.000607 | 0.000118 | 7.46E-05 |

| | | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|
| | SD | 0.000173 | 0.000572 | 0.00024 | 0.000479 | 0.000139 | 0.001279 |
| HDPE NONE | 0.4% HNO3 | -0.00062 | 7.25E-05 | -0.00054 | 0.001444 | 0.000226 | 0.001464 |
| | 0.4% HNO3 | -0.00159 | 0.002945 | -0.00033 | 0.000755 | 4.12E-05 | 0.00129 |
| | 0.4% HNO3 | 0.000817 | 0.000292 | -0.00029 | 0.002141 | 0.000196 | 0.001528 |
| | 0.4% HNO3 | 0.00035 | 0.000219 | -0.00083 | 0.001475 | 0.000123 | 0.000104 |
| | 0.4% HNO3 | -0.00035 | -0.0006 | -0.00062 | 0.001262 | 0.000144 | -0.00031 |
| | Mean | -0.00028 | 0.000585 | -0.00052 | 0.001415 | 0.000146 | 0.000815 |
| | SD | 0.000926 | 0.001366 | 0.000223 | 0.000498 | 7.14E-05 | 0.000856 |
| | 1 ml 8M | -0.00047 | -7.3E-05 | -0.00033 | -0.00033 | 0.000205 | -0.00034 |
| | 1 ml 8M | -0.00047 | 0.000183 | -0.00051 | -3E-05 | 0.000103 | -0.00069 |
| | 1 ml 8M | 6.32E-10 | -0.00022 | -0.00051 | -9E-05 | 8.19E-05 | -0.00062 |
| | 1 ml 8M | 0.000469 | 0.000621 | 0.000181 | 0.000328 | 8.18E-05 | -0.00062 |
| | 1 ml 8M | -0.00137 | 0.000201 | 0.000686 | -0.00018 | 1.35E-10 | -4.4E-05 |
| | Mean | -0.00037 | 0.000143 | -9.4E-05 | -6E-05 | 9.43E-05 | -0.00046 |
| | SD | 0.000682 | 0.000321 | 0.000519 | 0.000245 | 7.35E-05 | 0.00027 |
| FEP NONE | 0.4% HNO3 | 0.005956 | -0.0002 | 0.000469 | 0.000357 | -4.1E-05 | 0.005866 |
| | 0.4% HNO3 | 0.016121 | -0.00051 | 0.001047 | 0.001809 | 5.1E-05 | 0.003998 |
| | 0.4% HNO3 | 0.004908 | -0.00027 | 0.000794 | 0.000622 | 1.02E-05 | 0.004212 |
| | 0.4% HNO3 | 0.009012 | 0.000511 | 0.002056 | 0.003215 | 0.000163 | 0.030387 |
| | 0.4% HNO3 | 0.012285 | 0.000182 | 0.000649 | 0.004243 | 0.000294 | 0.006901 |
| | Mean | 0.009657 | -5.9E-05 | 0.001003 | 0.002049 | 9.54E-05 | 0.010273 |
| | SD | 0.00462 | 0.000405 | 0.000625 | 0.001668 | 0.000134 | 0.011308 |
| | 1 ml 8M | 0.612264 | 0.000855 | 0.005915 | 0.006741 | 0.000436 | 0.118801 |
| | 1 ml 8M | 0.023796 | -0.0004 | 0.002669 | 0.001206 | 0.000183 | 0.00465 |
| | 1 ml 8M | 0.027423 | 0.00029 | 0.001803 | 0.00094 | 0.000132 | 0.01395 |
| | 1 ml 8M | 0.132836 | 0.000888 | 0.00339 | 0.003317 | 0.000344 | 0.049857 |
| | 1 ml 8M | 0.024663 | 0.003346 | 0.003895 | 0.001935 | 3.04E-05 | 0.13444 |
| | Mean | 0.164197 | 0.000996 | 0.003534 | 0.002828 | 0.000225 | 0.06434 |

| | | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|
| | SD | 0.254773 | 0.001414 | 0.001546 | 0.002374 | 0.000164 | 0.059565 |
| HDPE PREWAS | 0.4% HNO3 | 0.000116 | -0.00047 | 0.000325 | 0.002842 | 6.07E-05 | 0.003068 |
| | 0.4% HNO3 | 0.001084 | -0.00063 | 0.000541 | 0.001785 | 9.1E-05 | 0.007475 |
| | 0.4% HNO3 | -0.00015 | -0.00034 | 0.000289 | 0.002076 | 4.04E-05 | 0.00286 |
| | 0.4% HNO3 | 0.001197 | -0.00013 | 0.001551 | 0.002775 | 0.000424 | 0.004083 |
| | 0.4% HNO3 | 0.001738 | 0.000126 | 0.000144 | 0.003061 | 0.000101 | 0.005517 |
| | Mean | 0.000796 | -0.00029 | 0.00057 | 0.002508 | 0.000143 | 0.004601 |
| | SD | 0.00079 | 0.000296 | 0.000567 | 0.000547 | 0.000159 | 0.00192 |
| | 1 ml 8M | 0.001932 | -0.00013 | 4.59E-10 | 0.001952 | 5.04E-05 | 0.001627 |
| | 1 ml 8M | 0.000464 | 0.000287 | 0.000215 | 0.000437 | 0.000212 | 0.002533 |
| | 1 ml 8M | 0.000465 | -0.00068 | 0.000538 | 0.001892 | -0.0001 | 0.003655 |
| | 1 ml 8M | -3.9E-05 | -0.00011 | -0.00025 | 0.00032 | 8.05E-05 | 0.004646 |
| | 1 ml 8M | -0.00105 | -0.00084 | 0.000716 | 0.001686 | 8.05E-05 | 0.00742 |
| | Mean | 0.000355 | -0.00029 | 0.000244 | 0.001258 | 6.45E-05 | 0.003976 |
| | SD | 0.001076 | 0.000462 | 0.000392 | 0.00081 | 0.000111 | 0.002237 |
| PETG EPA | 0.4% HNO3 | -0.00054 | -0.00045 | -0.00064 | 0.000727 | 2.01E-05 | 0.000704 |
| | 0.4% HNO3 | -0.00082 | -0.00127 | -0.00021 | -2.9E-05 | 0.000201 | 0.000977 |
| | 0.4% HNO3 | 7.77E-05 | -0.00047 | 0.000642 | 0.001626 | 0.000161 | 0.000919 |
| | 0.4% HNO3 | -0.00012 | -0.00072 | -0.00053 | 0.000783 | 0.000131 | 0.00102 |
| | 0.4% HNO3 | -0.00086 | -0.0005 | 0.000569 | 0.000551 | 0.00014 | 0.001364 |
| | Mean | -0.00045 | -0.00068 | -3.6E-05 | 0.000732 | 0.000131 | 0.000997 |
| | SD | 0.000417 | 0.000348 | 0.000607 | 0.000594 | 6.74E-05 | 0.000238 |
| | 1 ml 8M | 0.001671 | -5.4E-05 | 0.000249 | 0.011348 | 9.44E-12 | -0.0002 |
| | 1 ml 8M | 0.000893 | -9E-05 | 0.000391 | 0.010097 | -8E-05 | -0.00053 |
| | 1 ml 8M | 0.000698 | -0.0005 | -0.00032 | 0.006361 | 5.98E-05 | -0.0005 |
| | 1 ml 8M | -0.00058 | -0.00098 | -0.00011 | 0.006097 | 4.98E-05 | -0.00053 |
| | 1 ml 8M | 0.000232 | -0.0007 | 0.000639 | 0.005429 | 2.98E-05 | -0.00073 |
| | Mean | 0.000582 | -0.00046 | 0.000171 | 0.007867 | 1.19E-05 | -0.0005 |

|  |  |  | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|
|  |  | SD | 0.000832 | 0.000398 | 0.000385 | 0.002666 | 5.62E-05 | 0.000189 |
| PP | EPA | 0.4% HNO3 | -0.00023 | -0.00018 | 0.000284 | 0.000779 | 1.99E-05 | 0.000911 |
|  |  | 0.4% HNO3 | -7.7E-05 | 3.05E-10 | 0.000178 | 0.000837 | 0.000129 | 0.000753 |
|  |  | 0.4% HNO3 | -0.001 | -0.00016 | -0.00046 | 0.000317 | 0.000277 | 0.000781 |
|  |  | 0.4% HNO3 | -0.00069 | -8.9E-05 | 0.000533 | 0.00023 | 8.89E-05 | 0.000624 |
|  |  | 0.4% HNO3 | -0.00127 | -0.00039 | -0.00021 | 0.000921 | 0.000227 | 0.000609 |
|  |  | Mean | -0.00065 | -0.00016 | 6.39E-05 | 0.000617 | 0.000148 | 0.000736 |
|  |  | SD | 0.000503 | 0.000145 | 0.000398 | 0.000319 | 0.000104 | 0.000124 |
|  |  | 1 ml 8M | -0.00084 | -0.00053 | 1.51E-10 | 0.000144 | 6.9E-05 | -0.00102 |
|  |  | 1 ml 8M | 0.000842 | 0.000124 | -0.00021 | 0.002126 | 0.000226 | -0.00086 |
|  |  | 1 ml 8M | 7.66E-05 | 0.000124 | -7.1E-05 | 0.000115 | 0.000118 | -0.00068 |
|  |  | 1 ml 8M | 0.000191 | -0.00062 | 0.00032 | -0.0002 | 4.92E-05 | -0.00033 |
|  |  | 1 ml 8M | -0.00057 | -0.00048 | 0.000213 | 0.000143 | 7.88E-05 | -0.00117 |
|  |  | Mean | -6.2E-05 | -0.00028 | 4.98E-05 | 0.000465 | 0.000108 | -0.00081 |
|  |  | SD | 0.000666 | 0.00037 | 0.000216 | 0.00094 | 7.06E-05 | 0.000328 |
| HDPE | EPA | 0.4% HNO3 | 0.000344 | -0.00068 | 0.000356 | 0.000631 | 9.86E-05 | 0.000991 |
|  |  | 0.4% HNO3 | -0.0005 | -0.00032 | 0.00064 | 0.000201 | 0.000197 | 0.000297 |
|  |  | 0.4% HNO3 | 0.000764 | -0.001 | 0.000427 | 0.000573 | 9.86E-05 | 0.00034 |
|  |  | 0.4% HNO3 | -0.00057 | -0.00093 | -0.00061 | 0.000744 | 2.79E-11 | 0.001077 |
|  |  | 0.4% HNO3 | -0.00069 | -0.00039 | -0.00039 | 0.000801 | 2.79E-11 | 0.0018 |
|  |  | Mean | -0.00013 | -0.00066 | 8.53E-05 | 0.00059 | 7.89E-05 | 0.000901 |
|  |  | SD | 0.000645 | 0.000305 | 0.000548 | 0.000236 | 8.25E-05 | 0.000618 |
|  |  | 1 ml 8M | 3.81E-05 | -0.00075 | 0.000392 | 5.72E-05 | 0.000217 | 0.000241 |
|  |  | 1 ml 8M | -0.00023 | -0.00084 | -0.00061 | -0.00089 | 0.000158 | -0.0007 |
|  |  | 1 ml 8M | 0.000953 | 0.000445 | 0.000213 | 0.001312 | 0.000168 | -0.00079 |
|  |  | 1 ml 8M | 0.000648 | -8.9E-05 | -3.6E-05 | 0.001454 | 0.000207 | -0.00065 |
|  |  | 1 ml 8M | -0.00019 | -0.00055 | -0.00039 | -0.00023 | 0.000168 | -0.00099 |
|  |  | Mean | 0.000244 | -0.00036 | -8.5E-05 | 0.000342 | 0.000184 | -0.00058 |

| | | | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|
| | | SD | 0.00053 | 0.000533 | 0.000413 | 0.001012 | 2.67E-05 | 0.000476 |
| FEP | EPA | 0.4% HNO3 | 0.004999 | -0.00092 | 0.000817 | 0.000627 | 9.87E-05 | 0.000921 |
| | | 0.4% HNO3 | 0.002977 | -3.5E-05 | 0.000319 | 0.001338 | 7.9E-05 | 0.001005 |
| | | 0.4% HNO3 | 0.002902 | -0.00106 | -7.1E-05 | 0.001309 | 0.000158 | 0.001543 |
| | | 0.4% HNO3 | 0.00359 | -0.0008 | 0.000177 | 0.000484 | -4.7E-11 | 0.001967 |
| | | 0.4% HNO3 | 0.005233 | -0.00108 | 0.000142 | 0.000569 | 8.88E-05 | 0.001259 |
| | | Mean | 0.00394 | -0.00078 | 0.000277 | 0.000865 | 8.49E-05 | 0.001339 |
| | | SD | 0.001109 | 0.000432 | 0.000333 | 0.000422 | 5.65E-05 | 0.000427 |
| | | 1 ml 8M | 0.019256 | -0.00078 | 0.000602 | -0.00048 | -9.9E-06 | 0.001315 |
| | | 1 ml 8M | 0.016433 | -0.00057 | 0.000531 | 0.000739 | 8.88E-05 | 0.000311 |
| | | 1 ml 8M | 0.018272 | -0.00133 | 0.001203 | -0.00026 | 0.000128 | 0.006487 |
| | | 1 ml 8M | 0.015832 | -0.00065 | 0.001234 | 0.000992 | 9.85E-05 | 0.000721 |
| | | 1 ml 8M | 0.014991 | -0.00051 | 0.002358 | 0.000368 | 0.000177 | 0.000763 |
| | | Mean | 0.016957 | -0.00077 | 0.001185 | 0.000272 | 9.66E-05 | 0.001919 |
| | | SD | 0.001762 | 0.000328 | 0.000733 | 0.000631 | 6.87E-05 | 0.002578 |
| PETG | VIRGINIA | 0.4% HNO3 | 0.000115 | -0.00025 | 1.5E-10 | 0.002323 | -5.9E-05 | -0.00051 |
| | | 0.4% HNO3 | -0.00027 | -0.00062 | -0.00042 | -2.8E-05 | -0.00017 | -0.00059 |
| | | 0.4% HNO3 | -0.0008 | -0.00111 | -3.5E-05 | 0.001217 | 0.000118 | -0.00037 |
| | | 0.4% HNO3 | -0.00138 | -0.00067 | -0.00011 | 0.000226 | 0.000197 | -0.0005 |
| | | 0.4% HNO3 | 0.000191 | -0.00115 | -0.00059 | 0.000452 | 2.95E-05 | -0.00037 |
| | | Mean | -0.00043 | -0.00076 | -0.00023 | 0.000838 | 2.36E-05 | -0.00047 |
| | | SD | 0.000661 | 0.000376 | 0.000263 | 0.000952 | 0.000143 | 9.79E-05 |
| | | 1 ml 8M | 0.000383 | -0.00097 | 6.98E-05 | 0.004465 | 0.000167 | -0.00079 |
| | | 1 ml 8M | -0.00138 | -0.00122 | 0.000244 | 0.011242 | 0.000137 | -0.00071 |
| | | 1 ml 8M | 7.65E-05 | -0.00076 | 0.000348 | 0.007651 | 2.94E-05 | -0.00102 |
| | | 1 ml 8M | -0.00015 | -0.00131 | 0.000278 | 0.012981 | -9.8E-05 | -0.00048 |
| | | 1 ml 8M | 0.001034 | -0.00023 | 0.000139 | 0.011373 | 3.92E-05 | -0.00082 |
| | | Mean | -7.6E-06 | -0.0009 | 0.000216 | 0.009542 | 5.5E-05 | -0.00076 |

| | | | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|
| | | SD | 0.000886 | 0.00043 | 0.000111 | 0.003443 | 0.000104 | 0.000195 |
| PP | VIRGINIA | 0.4% HNO3 | -0.00019 | -0.00064 | 0.000139 | 0.001983 | 1.85E-11 | -0.00053 |
| | | 0.4% HNO3 | -0.00031 | -0.00014 | 0.000768 | 0.000426 | 2.78E-11 | 0.001605 |
| | | 0.4% HNO3 | -0.00054 | -0.00081 | -0.00028 | 0.000881 | -0.00014 | 0.001833 |
| | | 0.4% HNO3 | -0.00023 | -0.00078 | 7.45E-10 | 0.001167 | 0.000108 | 0.001408 |
| | | 0.4% HNO3 | 0.000268 | -0.00053 | 0.00028 | 0.000114 | 0.000147 | 0.001267 |
| | | Mean | -0.0002 | -0.00058 | 0.000182 | 0.000914 | 2.36E-05 | 0.001118 |
| | | SD | 0.000293 | 0.000269 | 0.000387 | 0.000722 | 0.000111 | 0.000943 |
| | | 1 ml 8M | 0.00023 | -0.00109 | -0.00056 | -0.00054 | 9.82E-06 | -0.00085 |
| | | 1 ml 8M | 0.00023 | -0.00078 | 0.000597 | -0.00037 | -2E-05 | -0.0011 |
| | | 1 ml 8M | -0.00172 | -0.00176 | -0.00011 | -0.00074 | 1.96E-05 | -0.00063 |
| | | 1 ml 8M | -0.00019 | -0.00042 | 0.000246 | 0.000201 | -9.8E-05 | -0.0008 |
| | | 1 ml 8M | -0.00096 | -0.00143 | -0.00021 | -0.0008 | 6.88E-05 | -0.00101 |
| | | Mean | -0.00048 | -0.0011 | -6.9E-06 | -0.00045 | -3.9E-06 | -0.00088 |
| | | SD | 0.000847 | 0.000527 | 0.000444 | 0.000403 | 6.16E-05 | 0.000185 |
| HDPE | VIRGINIA | 0.4% HNO3 | 4.65E-10 | -0.00083 | -0.00035 | 0.001581 | -8.8E-05 | 0.001641 |
| | | 0.4% HNO3 | -0.00054 | -0.00069 | 0.000528 | 0.001264 | 0.000128 | 0.001838 |
| | | 0.4% HNO3 | -0.00034 | -0.001 | -0.0006 | 0.000603 | 2.95E-05 | 0.00168 |
| | | 0.4% HNO3 | -0.00092 | -0.00143 | 0.000176 | 0.000517 | 2.78E-11 | 0.000768 |
| | | 0.4% HNO3 | -0.00119 | -0.00116 | 0.000597 | 0.001434 | 1.97E-05 | 0.001051 |
| | | Mean | -0.0006 | -0.00102 | 7.01E-05 | 0.00108 | 1.77E-05 | 0.001395 |
| | | SD | 0.000468 | 0.000288 | 0.00053 | 0.000489 | 7.72E-05 | 0.000461 |
| | | 1 ml 8M | -0.00088 | -0.00128 | 0.000175 | -0.00026 | 0.000118 | -0.00092 |
| | | 1 ml 8M | -0.00027 | -0.00091 | -0.00018 | -0.00075 | 9.83E-05 | -0.00096 |
| | | 1 ml 8M | -0.00023 | -0.00074 | -0.00056 | -0.00029 | 1.96E-05 | -0.00106 |
| | | 1 ml 8M | -0.00084 | -0.00095 | -0.00025 | -0.00069 | 4.91E-05 | -0.00074 |
| | | 1 ml 8M | -0.0008 | -0.00114 | 9.69E-10 | -0.00046 | -2E-05 | -0.00089 |
| | | Mean | -0.00061 | -0.00101 | -0.00016 | -0.00049 | 5.31E-05 | -0.00091 |

| | | | Mo 98 | Ag 107 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|
| | | SD | 0.000327 | 0.000212 | 0.000277 | 0.000224 | 5.63E-05 | 0.000119 |
| FEP | VIRGINIA | 0.4% HNO3 | -0.00084 | -0.00111 | -3.5E-05 | 0.000172 | 0.000177 | 0.000889 |
| | | 0.4% HNO3 | 0.001799 | -0.00049 | 0.000804 | 0.001771 | 0.000216 | 0.000986 |
| | | 0.4% HNO3 | -7.7E-05 | -0.00098 | 6.99E-05 | 0.001285 | 0.000186 | 0.001113 |
| | | 0.4% HNO3 | -3.8E-05 | -0.00099 | 0.000175 | 0.001141 | 3.92E-05 | 0.001423 |
| | | 0.4% HNO3 | -0.00054 | -0.00118 | -7E-05 | 2.85E-05 | 6.87E-05 | 0.001099 |
| | | Mean | 6.12E-05 | -0.00095 | 0.000189 | 0.000879 | 0.000137 | 0.001102 |
| | | SD | 0.001027 | 0.000269 | 0.000357 | 0.00075 | 7.83E-05 | 0.000201 |
| | | 1 ml 8M | 0.020135 | -0.00127 | 0.001049 | 8.55E-05 | -9.8E-05 | 0.001437 |
| | | 1 ml 8M | 0.020441 | -0.00076 | 0.000804 | -0.00037 | 5.88E-05 | 0.000972 |
| | | 1 ml 8M | 0.020824 | -0.00124 | 0.0007 | -0.00043 | 5.55E-11 | 0.001606 |
| | | 1 ml 8M | 0.023426 | -0.00127 | 0.001609 | -0.00065 | 0.000147 | 0.001677 |
| | | 1 ml 8M | 0.017263 | -0.00104 | 7E-05 | 0.000171 | 9.79E-05 | 0.001522 |
| | | Mean | 0.020418 | -0.00112 | 0.000846 | -0.00024 | 4.11E-05 | 0.001443 |
| | | SD | 0.002194 | 0.000221 | 0.000559 | 0.000353 | 9.46E-05 | 0.000278 |

# Bottle contaminant study for Se, Appendix 2.2

Solutions in the order laid out in Appendix 2.1

| | Se 77, ppb | Se 78, ppb |
|---|---|---|
| 1 | 0.000 | 0.001 |
| 2 | 0.000 | 0.000 |
| 3 | 0.000 | 0.001 |
| 4 | 0.000 | 0.001 |
| 5 | 0.000 | 0.000 |
| 6 | 0.000 | 0.000 |
| 7 | 0.000 | 0.000 |
| 8 | -0.001 | 0.000 |
| 9 | -0.001 | 0.000 |
| 10 | 0.000 | 0.000 |
| 11 | 0.001 | 0.000 |
| 12 | 0.001 | 0.000 |
| 13 | 0.001 | 0.001 |
| 14 | 0.000 | 0.000 |
| 15 | 0.001 | 0.000 |
| 16 | 0.000 | 0.000 |
| 17 | 0.000 | 0.000 |
| 18 | 0.000 | 0.000 |
| 19 | 0.000 | 0.000 |
| 20 | 0.000 | 0.000 |
| 21 | 0.000 | 0.000 |
| 22 | 0.000 | 0.000 |
| 23 | 0.000 | 0.000 |
| 24 | 0.000 | 0.000 |
| 25 | 0.000 | 0.000 |
| 26 | 0.000 | -0.001 |
| 27 | 0.000 | 0.000 |
| 28 | 0.000 | 0.000 |
| 29 | 0.001 | 0.000 |
| 30 | 0.000 | 0.000 |
| 31 | 0.000 | 0.000 |
| 32 | 0.000 | 0.000 |
| 33 | 0.000 | 0.000 |
| 34 | 0.000 | 0.001 |
| 35 | 0.000 | 0.000 |
| 36 | 0.000 | 0.001 |
| 37 | 0.000 | 0.001 |
| 38 | 0.000 | 0.000 |

| | | |
|---|---|---|
| 39 | 0.000 | 0.000 |
| 40 | 0.000 | 0.000 |
| 41 | 0.000 | 0.001 |
| 42 | 0.000 | 0.000 |
| 43 | 0.001 | 0.000 |
| 44 | 0.001 | 0.000 |
| 45 | 0.000 | 0.000 |
| 46 | 0.000 | 0.000 |
| 47 | 0.000 | 0.000 |
| 48 | 0.000 | 0.000 |
| 49 | 0.000 | 0.000 |
| 50 | 0.000 | 0.000 |
| 51 | 0.000 | 0.000 |
| 52 | 0.000 | 0.000 |
| 53 | 0.000 | 0.000 |
| 54 | -0.001 | 0.000 |
| 55 | -0.001 | -0.001 |
| 56 | 0.000 | -0.001 |
| 57 | -0.001 | -0.001 |
| 58 | 0.000 | 0.000 |
| 59 | 0.000 | -0.001 |
| 60 | 0.000 | -0.001 |
| 61 | 0.000 | 0.000 |
| 62 | 0.000 | 0.000 |
| 63 | 0.000 | 0.000 |
| 64 | 0.000 | 0.000 |
| 65 | 0.000 | 0.000 |
| 66 | 0.000 | 0.000 |
| 67 | 0.000 | 0.000 |
| 68 | 0.000 | 0.000 |
| 69 | 0.000 | 0.000 |
| 70 | 0.000 | 0.000 |
| 71 | 0.000 | 0.000 |
| 72 | 0.001 | 0.000 |
| 73 | 0.000 | -0.001 |
| 74 | 0.001 | 0.000 |
| 75 | 0.001 | 0.000 |
| 76 | 0.001 | 0.000 |
| 77 | 0.001 | 0.000 |
| 78 | 0.001 | 0.000 |
| 79 | 0.000 | 0.000 |
| 80 | 0.000 | 0.000 |

| | | |
|---|---|---|
| 81 | 0.000 | 0.000 |
| 82 | 0.000 | 0.000 |
| 83 | 0.000 | 0.000 |
| 84 | 0.000 | 0.000 |
| 85 | -0.001 | 0.000 |
| 86 | -0.001 | 0.000 |
| 87 | -0.001 | 0.000 |
| 88 | -0.001 | -0.001 |
| 89 | -0.001 | 0.000 |
| 90 | -0.001 | 0.000 |
| 91 | 0.000 | 0.001 |
| 92 | 0.000 | 0.000 |
| 93 | 0.000 | 0.000 |
| 94 | 0.000 | 0.000 |
| 95 | -0.001 | 0.000 |
| 96 | 0.000 | 0.000 |
| 97 | -0.001 | 0.000 |
| 98 | 0.000 | 0.000 |
| 99 | 0.000 | 0.000 |
| 100 | 0.000 | 0.000 |
| 101 | 0.000 | 0.000 |
| 102 | 0.000 | 0.000 |
| 103 | 0.000 | 0.000 |
| 104 | 0.000 | 0.000 |
| 105 | 0.000 | 0.000 |
| 106 | 0.000 | 0.000 |
| 107 | 0.000 | 0.000 |
| 108 | 0.000 | 0.000 |
| 109 | 0.000 | 0.000 |
| 110 | 0.000 | 0.000 |
| 111 | 0.000 | 0.000 |
| 112 | 0.000 | 0.000 |
| 113 | 0.000 | 0.000 |
| 114 | 0.000 | 0.000 |
| 115 | 0.000 | 0.000 |
| 116 | 0.000 | 0.000 |
| 117 | -0.001 | -0.001 |
| 118 | 0.000 | 0.000 |
| 119 | 0.000 | -0.001 |
| 120 | -0.001 | -0.001 |
| 121 | 0.001 | 0.000 |
| 122 | 0.000 | 0.000 |

| | | |
|---|---|---|
| 123 | 0.000 | 0.000 |
| 124 | 0.000 | 0.000 |
| 125 | 0.000 | 0.000 |
| 126 | 0.000 | 0.000 |
| 127 | 0.000 | 0.000 |
| 128 | 0.000 | 0.000 |
| 129 | 0.000 | 0.000 |
| 130 | 0.000 | 0.000 |

# Bottles contaminant study for Hg, Appendix 2.3

Notes: dichromate reagent has significant level of Hg in it. In EPA method 1638, PETG bottles are melted by 12M $HNO_3$ in water bath and in the oven at 60C they deform.
PP bottles become yellow and the HDPE slightly brittle.

| Bottle type | bottle # | Hg ppb .5% BrCl | bottle # | Hg ppb 2% HCl | bottle # | Hg ppb 0.04%,.2% $K_2Cr_2O_7$, $HNO_3$ |
|---|---|---|---|---|---|---|
| **EPA 1631** | | | | | | |
| FEP | 1 | -0.0003 | 21 | -0.0006 | 41 | 0.0035 |
| | 2 | -0.0002 | 22 | -0.0010 | 42 | 0.0024 |
| | 3 | -0.0006 | 23 | -0.0011 | 43 | 0.0026 |
| | 4 | -0.0009 | 24 | -0.0011 | 44 | 0.0022 |
| | 5 | -0.0008 | 25 | -0.0014 | 45 | 0.0021 |
| | Mean | -0.0006 | | -0.0010 | | 0.0026 |
| | SD | 0.0003 | | 0.0003 | | 0.0005 |
| HDPE | 6 | -0.0010 | 26 | -0.0015 | 46 | 0.0021 |
| | 7 | -0.0009 | 27 | -0.0016 | 47 | 0.0019 |
| | 8 | -0.0009 | 28 | -0.0009 | 48 | 0.0018 |
| | 9 | -0.0008 | 29 | -0.0005 | 49 | 0.0016 |
| | 10 | -0.0006 | 30 | -0.0009 | 50 | 0.0016 |
| | Mean | -0.0009 | | -0.0011 | | 0.0018 |
| | SD | 0.0002 | | 0.0005 | | 0.0002 |
| PETG | 11 | -0.0004 | 31 | -0.0009 | 51 | 0.0022 |
| | 12 | -0.0006 | 32 | -0.0008 | 52 | 0.0023 |
| | 13 | -0.0005 | 33 | -0.0008 | 53 | 0.0013 |
| | 14 | -0.0005 | 34 | -0.0005 | 54 | 0.0011 |
| | 15 | -0.0003 | 35 | -0.0006 | 55 | 0.0008 |
| | Mean | -0.0004 | | -0.0007 | | 0.0015 |
| | SD | 0.0001 | | 0.0002 | | 0.0007 |
| PP | 16 | -0.0005 | 36 | -0.0005 | 56 | 0.0006 |
| | 17 | -0.0006 | 37 | -0.0003 | 57 | 0.0005 |
| | 18 | -0.0006 | 38 | -0.0002 | 58 | 0.0006 |

| Bottle type | bottle # | Hg ppb .5% BrCl | bottle # | Hg ppb 2% HCl | bottle # | Hg ppb 0.04%,.2% K2Cr2O7, HNO3 |
|---|---|---|---|---|---|---|
| | 19 | 0.0006 | 39 | -0.0003 | 59 | 0.0006 |
| | 20 | -0.0005 | 40 | 0.0003 | 60 | 0.0009 |
| | Mean | -0.0003 | | -0.0002 | | 0.0006 |
| | SD | 0.000505643 | | 0.000287004 | | 0.000157041 |
| **EPA 1638** | | | | | | |
| FEP | 61 | 0.0001 | 81 | 0.0001 | 101 | 0.0020 |
| | 62 | 0.0000 | 82 | -0.0001 | 102 | 0.0015 |
| | 63 | -0.0001 | 83 | -0.0001 | 103 | 0.0024 |
| | 64 | -0.0003 | 84 | -0.0001 | 104 | 0.0016 |
| | 65 | -0.0003 | 85 | -0.0001 | 105 | 0.0016 |
| | Mean | -0.0001 | | -0.0001 | | 0.0018 |
| | SD | 0.0002 | | 0.0001 | | 0.0004 |
| HDPE | 66 | -0.0001 | 86 | -0.0003 | 106 | 0.0016 |
| | 67 | -0.0001 | 87 | -0.0004 | 107 | 0.0018 |
| | 68 | -0.0002 | 88 | -0.0003 | 108 | 0.0015 |
| | 69 | -0.0002 | 89 | -0.0003 | 109 | 0.0018 |
| | 70 | -0.0001 | 90 | -0.0004 | 110 | 0.0020 |
| | Mean | -0.0001 | | -0.0003 | | 0.0017 |
| | SD | 0.0001 | | 0.0000 | | 0.0002 |
| PETG | 71 | 0.0001 | 91 | 0.0008 | 111 | 0.0014 |
| | 72 | 0.0002 | 92 | 0.0005 | 112 | 0.0012 |
| | 73 | 0.0002 | 93 | 0.0006 | 113 | 0.0007 |
| | 74 | 0.0003 | 94 | 0.0004 | 114 | 0.0005 |
| | 75 | 0.0000 | 95 | 0.0005 | 115 | 0.0007 |
| | Mean | 0.0002 | | 0.0006 | | 0.0009 |
| | SD | 0.0001 | | 0.0001 | | 0.0004 |
| PP | 76 | 0.0000 | 96 | 0.0003 | 116 | 0.0005 |

## Bottles contaminant study for Hg, Appendix 2.3

| Bottle type | bottle # | Hg ppb .5% BrCl | bottle # | Hg ppb 2% HCl | bottle # | Hg ppb 0.04%,.2% K2Cr2O7, HNO3 |
|---|---|---|---|---|---|---|
| | 77 | 0.0002 | 97 | 0.0002 | 117 | 0.0007 |
| | 78 | 0.0000 | 98 | 0.0001 | 118 | 0.0004 |
| | 79 | -0.0001 | 99 | 0.0001 | 119 | 0.0007 |
| | 80 | 0.0000 | 100 | 0.0000 | 120 | 0.0007 |
| | Mean | 0.0000 | | 0.0002 | | 0.0006 |
| | SD | 9.89544E-05 | | 0.000121034 | | 0.000125667 |
| **No cleaning** | | | | | | |
| FEP | 121 | 0.0010 | 141 | -0.0005 | 161 | 0.0015 |
| | 122 | 0.0007 | 142 | 0.0006 | 162 | 0.0009 |
| | 123 | 0.0001 | 143 | 0.0002 | 163 | 0.0027 |
| | 124 | 0.0009 | 144 | 0.0002 | 164 | 0.0008 |
| | 125 | 0.0009 | 145 | 0.0007 | 165 | 0.0025 |
| | Mean | 0.0007 | | 0.0002 | | 0.0017 |
| | SD | 0.0004 | | 0.0005 | | 0.0009 |
| HDPE | 126 | -0.0001 | 146 | -0.0004 | 166 | 0.0013 |
| | 127 | -0.0001 | 147 | 0.0009 | 167 | 0.0021 |
| | 128 | 0.0000 | 148 | 0.0005 | 168 | 0.0015 |
| | 129 | -0.0006 | 149 | 0.0004 | 169 | 0.0016 |
| | 130 | -0.0006 | 150 | 0.0005 | 170 | 0.0012 |
| | Mean | -0.0003 | | 0.0004 | | 0.0015 |
| | SD | 0.0003 | | 0.0005 | | 0.0004 |
| PETG | 131 | -0.0005 | 151 | -0.0005 | 171 | 0.0010 |
| | 132 | -0.0003 | 152 | 0.0004 | 172 | 0.0014 |
| | 133 | -0.0006 | 153 | 0.0002 | 173 | 0.0011 |
| | 134 | -0.0006 | 154 | 0.0001 | 174 | 0.0011 |
| | 135 | -0.0005 | 155 | 0.0000 | 175 | 0.0009 |
| | Mean | -0.0005 | | 0.0001 | | 0.0011 |
| | SD | 0.0001 | | 0.0003 | | 0.0002 |

| Bottle type | bottle # | Hg ppb .5% BrCl | bottle # | Hg ppb 2% HCl | bottle # | Hg ppb 0.04%,.2% K2Cr2O7, HNO3 |
|---|---|---|---|---|---|---|
| PP | 136 | -0.0003 | 156 | -0.0006 | 176 | 0.0014 |
| | 137 | -0.0001 | 157 | 0.0002 | 177 | 0.0005 |
| | 138 | -0.0001 | 158 | 0.0004 | 178 | 0.0006 |
| | 139 | -0.0003 | 159 | 0.0002 | 179 | 0.0025 |
| | 140 | 0.0003 | 160 | 0.0003 | 180 | 0.0019 |
| | Mean | -0.0001 | | 0.0001 | | 0.0014 |
| | SD | 0.0002 | | 0.0004 | | 0.0008 |
| HDPE Preclean | 181 | 0.0001 | 186 | 0.0000 | 191 | 0.0015 |
| | 182 | 0.0002 | 187 | 0.0005 | 192 | 0.0015 |
| | 183 | 0.0004 | 188 | 0.0005 | 193 | 0.0013 |
| | 184 | 0.0004 | 189 | 0.0003 | 194 | 0.0011 |
| | 185 | 0.0008 | 190 | 0.0002 | 195 | 0.0010 |
| | Mean | 0.0004 | | 0.0003 | | 0.0013 |
| | SD | 0.000271324 | | 0.000224499 | | 0.000233874 |

| ***All*** | ***values*** | ***in*** | ***ppb*** | ***except*** | ***Hg, Se*** | | | |
|---|---|---|---|---|---|---|---|---|
| **Manufact.** | **Descript.** | **Part#** | **Size** | **Type** | **Sample** | **Al 27** | **Cr 52** | **Fe 54** |
| Gelman Sciences | Ion Chromatography Acrodisc | 4585 | 0.45 | Syringe filter | D.I. | -0.008 | 0.006 | -0.030 |
| | | | | | | -0.008 | 0.014 | -1.074 |
| | | | | | | 0.005 | 0.000 | 1.282 |
| | | | | | Mean | -0.004 | 0.007 | 0.059 |
| | | | | | SD | 0.007 | 0.007 | 1.181 |
| | | | | | .4%HNO3 | 0.034 | -0.010 | 0.974 |
| | | | | | | 0.015 | -0.011 | -1.127 |
| | | | | | | 0.011 | -0.015 | 1.415 |
| | | | | | Mean | 0.020 | -0.012 | 0.421 |
| | | | | | SD | 0.012 | 0.002 | 1.358 |
| Gelman Sciences | Nylon Acrodisc GF | 4549 | 0.45 | Syringe filter | D.I. | 0.057 | -0.002 | -1.630 |
| | | | | | | 0.070 | -0.003 | 4.641 |
| | | | | | Mean | 0.063 | -0.003 | 1.506 |
| | | | | | SD | 0.010 | 0.001 | 4.435 |
| | | | | | .4%HNO3 | 26.197 | 0.031 | 5.339 |
| | | | | | | 25.861 | 0.044 | 6.698 |
| | | | | | Mean | 26.029 | 0.037 | 6.018 |
| | | | | | SD | 0.238 | 0.009 | 0.961 |
| Gelman Sciences | GHP Acrodisc | 4560 | 0.45 | Syringe filter | D.I. | 0.109 | -0.003 | -0.670 |
| | | | | | | 0.013 | -0.028 | 1.422 |
| | | | | | | 0.045 | -0.008 | -0.277 |
| | | | | | Mean | 0.056 | -0.013 | 0.158 |
| | | | | | SD | 0.049 | 0.013 | 1.112 |
| | | | | | .4%HNO3 | 0.041 | -0.011 | 2.659 |
| | | | | | | 0.056 | -0.002 | 4.234 |
| | | | | | | 0.060 | -0.006 | 3.502 |
| | | | | | Mean | 0.052 | -0.006 | 3.465 |
| | | | | | SD | 0.010 | 0.004 | 0.788 |
| Millipore | Sterivex syringe filter capsules | SVHVL 10 15 | 0.45 | Syringe filter | D.I. | 0.161 | -0.011 | -2.549 |
| | | | | | | 0.338 | 0.012 | 0.556 |
| | | | | | | 0.089 | -0.024 | -0.982 |
| | | | | | Mean | 0.196 | -0.008 | -0.992 |
| | | | | | SD | 0.128 | 0.018 | 1.553 |
| | | | | | .4%HNO3 | 0.042 | 0.013 | -3.331 |
| | | | | | | 0.094 | 0.010 | 0.722 |
| | | | | | | 0.107 | -0.002 | -1.404 |
| | | | | | Mean | 0.081 | 0.007 | -1.338 |
| | | | | | SD | 0.035 | 0.008 | 2.027 |
| **Manufact.** | **Descript.** | **Part#** | **Size** | **Type** | **Sample** | **Al 27** | **Cr 52** | **Fe 54** |
| Millipore | Millex-HV filter unit | SLHV 025NS | 0.45 | Syringe filter | D.I. | -0.001 | 0.007 | 2.197 |
| | | | | | | 0.028 | 0.005 | 0.526 |
| | | | | | | 0.005 | -0.016 | 0.565 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | Mean | 0.011 | -0.002 | 1.096 |
| | | | | | SD | 0.015 | 0.013 | 0.953 |
| | | | | | .4%HNO3 | 3.561 | -0.009 | 1.664 |
| | | | | | | 0.101 | -0.001 | 0.453 |
| | | | | | | 0.050 | -0.002 | -1.124 |
| | | | | | Mean | 1.238 | -0.004 | 0.331 |
| | | | | | SD | 2.012 | 0.005 | 1.398 |
| Millipore | Millex-LS Prefilter or Milles-SV | SLLS 025NS or SLSV | 5 | Syringe filter | D.I. | 0.011 | 0.001 | 2.799 |
| | | | | | | -0.005 | 0.005 | -0.595 |
| | | | | | | -0.009 | -0.001 | 1.247 |
| | | | | | Mean | -0.001 | 0.002 | 1.150 |
| | | | | | SD | 0.011 | 0.003 | 1.699 |
| | | | | | .4%HNO3 | 0.019 | 0.000 | 4.471 |
| | | | | | | 0.012 | 0.003 | 1.124 |
| | | | | | | 0.035 | 0.008 | 2.399 |
| | | | | | Mean | 0.022 | 0.004 | 2.665 |
| | | | | | SD | 0.012 | 0.004 | 1.689 |
| Millipore | Millex HN | SLHN 025NS | 0.45 | Syringe filter | D.I. | 0.022 | 0.016 | 4.138 |
| | | | | | | -0.012 | -0.001 | -0.186 |
| | | | | | | 0.010 | 0.004 | 5.741 |
| | | | | | Mean | 0.007 | 0.007 | 3.231 |
| | | | | | SD | 0.017 | 0.009 | 3.066 |
| | | | | | .4%HNO3 | 0.020 | 0.010 | 1.536 |
| | | | | | | 0.027 | -0.007 | 3.842 |
| | | | | | | 0.091 | -0.006 | 2.842 |
| | | | | | Mean | 0.046 | -0.001 | 2.740 |
| | | | | | SD | 0.039 | 0.009 | 1.156 |
| Millipore | Millicup bottle top filter | SJHV M47 10 | 0.45 | Vacuum filter | D.I. | 0.053 | 0.095 | 0.850 |
| | | | | | | 0.105 | 0.007 | 0.963 |
| | | | | | | 0.033 | 0.009 | 3.137 |
| | | | | | Mean | 0.064 | 0.037 | 1.650 |
| | | | | | SD | 0.037 | 0.051 | 1.289 |
| | | | | | R+D.I. | -0.001 | 0.004 | 4.136 |
| | | | | | | -0.004 | 0.008 | 2.512 |
| | | | | | | 0.019 | 0.012 | 5.832 |
| | | | | | Mean | 0.004 | 0.008 | 4.160 |
| | | | | | SD | 0.012 | 0.004 | 1.660 |
| **Manufact.** | **Descript.** | **Part#** | **Size** | **Type** | **Sample** | **Al 27** | **Cr 52** | **Fe 54** |
| | | | | | .4%HNO3 | 0.190 | 0.019 | 3.643 |
| | | | | | | 0.113 | 0.019 | 2.367 |
| | | | | | | 0.098 | 0.007 | 2.794 |
| | | | | | Mean | 0.134 | 0.015 | 2.934 |
| | | | | | SD | 0.049 | 0.007 | 0.650 |
| Falcon | Falcon | 7104 | 0.45 | Vacuum | D.I. | 0.035 | 0.022 | 2.017 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | bottle top | | | filter | | 0.040 | 0.020 | 2.858 |
| | | | | | | 0.280 | 0.014 | -1.508 |
| | | | | | Mean | 0.118 | 0.018 | 1.122 |
| | | | | | SD | 0.140 | 0.004 | 2.316 |
| | | | | | R+D.I. | -0.005 | 0.008 | 4.593 |
| | | | | | | -0.015 | -0.007 | 2.087 |
| | | | | | | -0.008 | 0.005 | 3.643 |
| | | | | | Mean | -0.009 | 0.002 | 3.441 |
| | | | | | SD | 0.005 | 0.008 | 1.265 |
| | | | | | .4%HNO3 | 0.071 | -0.007 | 1.707 |
| | | | | | | 0.138 | 0.013 | -2.562 |
| | | | | | | 0.050 | -0.007 | 1.999 |
| | | | | | Mean | 0.086 | 0.000 | 0.381 |
| | | | | | SD | 0.046 | 0.011 | 2.553 |
| Millipore | All glass filtration unit | XX15 047 00 ıembrane HAWP 047 00 | 0.45 | Vacuum filter | D.I. | 0.606 | 0.003 | 3.466 |
| | | | | | | 0.083 | 0.003 | 0.374 |
| | | | | | | 0.315 | -0.003 | -1.611 |
| | | | | | Mean | 0.335 | 0.001 | 0.743 |
| | | | | | SD | 0.262 | 0.003 | 2.559 |
| | | | | | R+D.I. | 0.294 | 0.001 | -3.049 |
| | | | | | | 0.003 | -0.007 | 0.719 |
| | | | | | | 0.450 | -0.001 | -4.257 |
| | | | | | Mean | 0.249 | -0.002 | -2.196 |
| | | | | | SD | 0.227 | 0.004 | 2.595 |
| | | | | | .4%HNO3 | 0.924 | -0.001 | -2.453 |
| | | | | | | 0.847 | -0.008 | -0.687 |
| | | | | | | 0.898 | -0.003 | -3.456 |
| | | | | | Mean | 0.890 | -0.004 | -2.199 |
| | | | | | SD | 0.039 | 0.004 | 1.402 |
| | As above | | 5 | | D.I. | 0.044 | 0.003 | 1.982 |
| | | | | | R+D.I. | 0.260 | 0.003 | 2.696 |
| | | | | | .4%HNO3 | 0.465 | 0.008 | 2.296 |

| **Manufact.** | **Descript.** | **Part#** | **Size** | **Type** | **Sample** | **Al 27** | **Cr 52** | **Fe 54** |
|---|---|---|---|---|---|---|---|---|
| Gelman Sciences | Groundwater sampling capsule | 12175 or 12176 | 0.45 | Inline filter | D.I. | 0.607 | 0.114 | 2.970 |
| | | | | | | 0.593 | 0.062 | -0.623 |
| | | | | | | 0.533 | 0.047 | 2.416 |
| | | | | | Mean | 0.578 | 0.074 | 1.588 |
| | | | | | SD | 0.040 | 0.035 | 1.935 |
| | | | | | R+D.I. | 0.080 | 0.024 | 4.042 |
| | | | | | | 0.042 | 0.015 | 4.817 |
| | | | | | | 0.014 | 0.041 | -0.632 |
| | | | | | Mean | 0.046 | 0.027 | 2.743 |
| | | | | | SD | 0.033 | 0.013 | 2.948 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | .4%HNO3 | 1.143 | 0.041 | 3.189 |
| | | | | | | 1.389 | 0.033 | 6.041 |
| | | | | | | 1.224 | 0.030 | 1.045 |
| | | | | | Mean | 1.252 | 0.035 | 3.425 |
| | | | | | SD | 0.125 | 0.006 | 2.506 |
| Gelman Sciences | Aquaprep | 4270 | 0.45 | Inline filter | D.I. | -0.002 | -0.004 | -0.306 |
| | | | | | | -0.019 | 0.003 | -6.487 |
| | | | | | | -0.019 | 0.021 | -4.037 |
| | | | | | Mean | -0.013 | 0.007 | -3.610 |
| | | | | | SD | 0.010 | 0.013 | 3.112 |
| | | | | | R+D.I. | -0.008 | 0.008 | -1.033 |
| | | | | | | -0.011 | 0.010 | 0.807 |
| | | | | | | -0.017 | 0.015 | 0.422 |
| | | | | | Mean | -0.012 | 0.011 | 0.066 |
| | | | | | SD | 0.005 | 0.003 | 0.970 |
| | | | | | .4%HNO3 | 0.084 | 0.006 | 2.219 |
| | | | | | | 0.123 | -0.002 | 2.678 |
| | | | | | | 0.023 | 0.004 | -1.829 |
| | | | | | Mean | 0.077 | 0.003 | 1.022 |
| | | | | | SD | 0.050 | 0.004 | 2.480 |
| Gelman Sciences | Aquaprep 250 | 12026 or 12027 | 0.45 | Inline filter | D.I. | 0.144 | 0.021 | 1.029 |
| | | | | | | 0.103 | 0.004 | 3.702 |
| | | | | | | 0.125 | -0.002 | -0.556 |
| | | | | | Mean | 0.124 | 0.008 | 1.392 |
| | | | | | SD | 0.021 | 0.012 | 2.152 |
| | | | | | R+D.I. | 0.012 | -0.006 | 0.083 |
| | | | | | | 0.017 | 0.003 | 0.205 |
| | | | | | | 0.040 | -0.002 | 0.633 |
| | | | | | Mean | 0.023 | -0.002 | 0.307 |
| | | | | | SD | 0.015 | 0.005 | 0.289 |
| **Manufact.** | **Descript.** | **Part#** | **Size** | **Type** | **Sample** | **Al 27** | **Cr 52** | **Fe 54** |
| | | | | | .4%HNO3 | 0.894 | 0.002 | 2.006 |
| | | | | | | 1.078 | 0.017 | 2.641 |
| | | | | | | 1.054 | 0.002 | 3.966 |
| | | | | | Mean | 1.009 | 0.007 | 2.871 |
| | | | | | SD | 0.100 | 0.009 | 1.000 |

| Type | Sample | Mn 55 | Co 59 | Ni 60 | Ni 62 | Cu 63 | Cu 65 |
|---|---|---|---|---|---|---|---|
| Syringe filter | D.I. | 0.011 | 0.000 | 0.048 | 0.052 | -0.004 | -0.009 |
| | | 0.015 | 0.000 | 0.054 | 0.057 | -0.015 | -0.021 |
| | | 0.014 | 0.000 | 0.054 | 0.036 | -0.013 | -0.018 |
| | Mean | 0.014 | 0.000 | 0.052 | 0.048 | -0.011 | -0.016 |
| | SD | 0.002 | 0.000 | 0.003 | 0.011 | 0.006 | 0.007 |
| | .4%HNO3 | 0.004 | -0.001 | 0.017 | 0.003 | -0.012 | -0.016 |
| | | 0.001 | -0.001 | 0.013 | 0.020 | 0.006 | 0.003 |
| | | 0.002 | -0.001 | 0.016 | -0.016 | -0.016 | -0.019 |
| | Mean | 0.003 | -0.001 | 0.015 | 0.002 | -0.007 | -0.011 |
| | SD | 0.002 | 0.000 | 0.003 | 0.018 | 0.012 | 0.012 |
| Syringe filter | D.I. | 0.014 | -0.001 | 0.013 | 0.035 | 0.062 | 0.075 |
| | | 0.017 | -0.001 | 0.022 | 0.015 | 0.080 | 0.086 |
| | Mean | 0.015 | -0.001 | 0.018 | 0.025 | 0.071 | 0.081 |
| | SD | 0.002 | 0.000 | 0.006 | 0.014 | 0.013 | 0.008 |
| | .4%HNO3 | 0.077 | -0.001 | 0.024 | 0.022 | 1.188 | 1.265 |
| | | 0.080 | 0.000 | 0.023 | -0.008 | 1.841 | 1.899 |
| | Mean | 0.078 | -0.001 | 0.023 | 0.007 | 1.514 | 1.582 |
| | SD | 0.002 | 0.000 | 0.001 | 0.021 | 0.462 | 0.448 |
| Syringe filter | D.I. | 0.019 | 0.000 | 0.050 | 0.028 | 0.006 | 0.001 |
| | | 0.014 | 0.000 | 0.032 | 0.004 | -0.005 | -0.007 |
| | | 0.017 | 0.000 | 0.049 | 0.053 | 0.000 | -0.005 |
| | Mean | 0.017 | 0.000 | 0.044 | 0.029 | 0.000 | -0.004 |
| | SD | 0.003 | 0.000 | 0.010 | 0.024 | 0.005 | 0.004 |
| | .4%HNO3 | 0.021 | -0.001 | 0.010 | -0.033 | -0.010 | -0.012 |
| | | 0.022 | -0.001 | 0.008 | -0.051 | -0.015 | -0.019 |
| | | 0.020 | -0.001 | 0.013 | -0.011 | 0.002 | -0.001 |
| | Mean | 0.021 | -0.001 | 0.010 | -0.032 | -0.008 | -0.011 |
| | SD | 0.001 | 0.000 | 0.002 | 0.020 | 0.009 | 0.009 |
| Syringe filter | D.I. | 0.009 | 0.007 | 0.011 | 0.000 | -0.019 | -0.022 |
| | | 0.011 | 0.006 | 0.007 | -0.006 | -0.007 | -0.013 |
| | | 0.002 | 0.002 | 0.006 | 0.001 | -0.024 | -0.024 |
| | Mean | 0.007 | 0.005 | 0.008 | -0.002 | -0.017 | -0.020 |
| | SD | 0.005 | 0.003 | 0.003 | 0.004 | 0.008 | 0.006 |
| | .4%HNO3 | 0.008 | 0.008 | 0.004 | 0.007 | -0.009 | -0.012 |
| | | 0.009 | 0.010 | 0.008 | 0.016 | 0.213 | 0.215 |
| | | 0.009 | 0.008 | 0.010 | 0.008 | 0.010 | 0.006 |
| | Mean | 0.009 | 0.009 | 0.007 | 0.010 | 0.071 | 0.070 |
| | SD | 0.001 | 0.001 | 0.003 | 0.005 | 0.123 | 0.126 |
| **Type** | **Sample** | **Mn 55** | **Co 59** | **Ni 60** | **Ni 62** | **Cu 63** | **Cu 65** |
| Syringe filter | D.I. | 0.001 | -0.001 | 0.000 | -0.010 | -0.014 | -0.020 |
| | | 0.002 | -0.001 | 0.005 | 0.023 | 0.192 | 0.198 |
| | | 0.000 | -0.001 | 0.008 | -0.008 | -0.008 | -0.011 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Mean | 0.001 | -0.001 | 0.004 | 0.002 | 0.056 | 0.056 |
| | SD | 0.001 | 0.000 | 0.004 | 0.018 | 0.117 | 0.123 |
| | .4%HNO3 | 0.001 | -0.001 | 0.010 | 0.012 | -0.013 | -0.013 |
| | | 0.002 | -0.001 | 0.019 | 0.020 | 0.056 | 0.053 |
| | | 0.001 | -0.001 | 0.021 | 0.020 | -0.007 | -0.009 |
| | Mean | 0.002 | -0.001 | 0.016 | 0.017 | 0.012 | 0.010 |
| | SD | 0.001 | 0.000 | 0.006 | 0.005 | 0.038 | 0.037 |
| Syringe filter | D.I. | 0.002 | -0.001 | 0.008 | -0.008 | -0.004 | -0.010 |
| | | 0.001 | -0.001 | 0.008 | 0.024 | -0.012 | -0.015 |
| | | 0.001 | -0.001 | 0.007 | 0.020 | -0.022 | -0.024 |
| | Mean | 0.002 | -0.001 | 0.008 | 0.012 | -0.013 | -0.016 |
| | SD | 0.000 | 0.000 | 0.000 | 0.017 | 0.009 | 0.007 |
| | .4%HNO3 | 0.002 | -0.001 | 0.009 | -0.028 | 0.008 | 0.005 |
| | | 0.002 | -0.001 | 0.014 | 0.015 | 0.012 | 0.009 |
| | | 0.003 | -0.001 | 0.017 | -0.003 | 0.018 | 0.018 |
| | Mean | 0.002 | -0.001 | 0.013 | -0.005 | 0.013 | 0.010 |
| | SD | 0.001 | 0.000 | 0.004 | 0.022 | 0.005 | 0.007 |
| Syringe filter | D.I. | 0.002 | -0.001 | 0.012 | -0.014 | -0.009 | -0.014 |
| | | 0.000 | -0.001 | 0.011 | -0.001 | -0.014 | -0.016 |
| | | 0.002 | -0.001 | 0.008 | -0.033 | 0.000 | -0.006 |
| | Mean | 0.001 | -0.001 | 0.010 | -0.016 | -0.008 | -0.012 |
| | SD | 0.001 | 0.000 | 0.002 | 0.016 | 0.007 | 0.005 |
| | .4%HNO3 | 0.000 | 0.002 | 0.006 | 0.006 | -0.015 | -0.019 |
| | | 0.000 | -0.001 | 0.018 | -0.024 | -0.012 | -0.011 |
| | | 0.001 | -0.001 | 0.014 | 0.000 | 0.021 | 0.021 |
| | Mean | 0.001 | 0.000 | 0.013 | -0.006 | -0.002 | -0.003 |
| | SD | 0.000 | 0.002 | 0.006 | 0.016 | 0.020 | 0.022 |
| Vacuum filter | D.I. | 0.016 | 0.001 | 0.088 | 0.100 | 0.000 | -0.004 |
| | | 0.002 | 0.009 | 0.013 | 0.046 | -0.003 | -0.006 |
| | | 0.002 | -0.001 | 0.005 | -0.001 | -0.004 | -0.010 |
| | Mean | 0.007 | 0.003 | 0.035 | 0.048 | -0.002 | -0.007 |
| | SD | 0.008 | 0.005 | 0.046 | 0.051 | 0.002 | 0.003 |
| | R+D.I. | 0.001 | -0.001 | 0.003 | -0.011 | -0.016 | -0.020 |
| | | 0.001 | -0.001 | 0.004 | 0.025 | -0.016 | -0.019 |
| | | 0.002 | -0.001 | 0.003 | 0.001 | 0.008 | 0.004 |
| | Mean | 0.001 | -0.001 | 0.004 | 0.005 | -0.008 | -0.012 |
| | SD | 0.001 | 0.000 | 0.000 | 0.018 | 0.014 | 0.014 |
| **Type** | **Sample** | **Mn 55** | **Co 59** | **Ni 60** | **Ni 62** | **Cu 63** | **Cu 65** |
| | .4%HNO3 | 0.004 | -0.001 | 0.023 | 0.016 | 0.036 | 0.028 |
| | | 0.003 | -0.001 | 0.012 | 0.027 | 0.040 | 0.036 |
| | | 0.004 | 0.000 | 0.012 | 0.026 | 0.015 | 0.009 |
| | Mean | 0.004 | -0.001 | 0.016 | 0.023 | 0.031 | 0.025 |
| | SD | 0.001 | 0.001 | 0.007 | 0.006 | 0.013 | 0.014 |
| Vacuum | D.I. | 0.056 | -0.001 | 0.006 | 0.046 | 0.039 | 0.038 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| filter | | 0.047 | 0.000 | 0.001 | 0.009 | -0.003 | -0.007 |
| | | 0.045 | -0.001 | 0.005 | 0.029 | 0.048 | 0.042 |
| | Mean | 0.049 | -0.001 | 0.004 | 0.028 | 0.028 | 0.025 |
| | SD | 0.006 | 0.000 | 0.003 | 0.019 | 0.027 | 0.027 |
| | R+D.I. | 0.002 | -0.001 | 0.005 | -0.008 | 0.004 | 0.000 |
| | | 0.003 | -0.001 | 0.002 | 0.006 | -0.024 | -0.025 |
| | | 0.005 | -0.001 | 0.003 | 0.008 | -0.004 | -0.009 |
| | Mean | 0.003 | -0.001 | 0.003 | 0.002 | -0.008 | -0.011 |
| | SD | 0.001 | 0.000 | 0.002 | 0.009 | 0.014 | 0.012 |
| | .4%HNO3 | 0.009 | -0.001 | 0.006 | 0.007 | 0.014 | 0.012 |
| | | 0.008 | -0.001 | -0.002 | 0.048 | -0.006 | -0.009 |
| | | 0.007 | -0.001 | 0.000 | 0.030 | -0.016 | -0.018 |
| | Mean | 0.008 | -0.001 | 0.002 | 0.029 | -0.003 | -0.005 |
| | SD | 0.001 | 0.000 | 0.004 | 0.020 | 0.015 | 0.016 |
| Vacuum filter | D.I. | 0.006 | 0.001 | 0.020 | | | 0.012 |
| | | 0.003 | 0.000 | -0.017 | | | 0.014 |
| | | 0.003 | 0.000 | -0.022 | | | 0.028 |
| | Mean | 0.004 | 0.000 | -0.006 | | | 0.018 |
| | SD | 0.002 | 0.001 | 0.023 | | | 0.009 |
| | R+D.I. | 0.002 | -0.001 | 0.007 | | | 0.083 |
| | | -0.001 | 0.000 | -0.013 | | | 0.012 |
| | | 0.002 | 0.000 | 0.000 | | | 0.059 |
| | Mean | 0.001 | 0.000 | -0.002 | | | 0.051 |
| | SD | 0.002 | 0.000 | 0.010 | | | 0.036 |
| | .4%HNO3 | 0.011 | 0.000 | 0.018 | | | 0.493 |
| | | 0.011 | 0.000 | 0.002 | | | 0.314 |
| | | 0.016 | -0.001 | -0.002 | | | 0.388 |
| | Mean | 0.013 | 0.000 | 0.006 | | | 0.398 |
| | SD | 0.003 | 0.000 | 0.011 | | | 0.090 |
| | D.I. | 0.002 | -0.001 | 0.012 | 0.004 | 0.033 | 0.028 |
| | R+D.I. | 0.001 | -0.001 | 0.012 | -0.006 | -0.003 | -0.006 |
| | .4%HNO3 | 0.008 | 0.000 | 0.029 | 0.013 | 0.269 | 0.250 |

| **Type** | **Sample** | **Mn 55** | **Co 59** | **Ni 60** | **Ni 62** | **Cu 63** | **Cu 65** |
|---|---|---|---|---|---|---|---|
| Inline filter | D.I. | 0.039 | 0.001 | 0.137 | 0.162 | 0.367 | 0.364 |
| | | 0.023 | 0.001 | 0.066 | 0.100 | 0.184 | 0.181 |
| | | 0.029 | 0.001 | 0.074 | 0.105 | 0.169 | 0.165 |
| | Mean | 0.030 | 0.001 | 0.092 | 0.123 | 0.240 | 0.237 |
| | SD | 0.008 | 0.000 | 0.039 | 0.034 | 0.110 | 0.111 |
| | R+D.I. | 0.002 | -0.001 | 0.005 | -0.011 | -0.010 | -0.015 |
| | | 0.008 | -0.001 | 0.010 | 0.007 | -0.010 | -0.014 |
| | | 0.003 | -0.001 | 0.005 | 0.049 | -0.022 | -0.026 |
| | Mean | 0.004 | -0.001 | 0.007 | 0.015 | -0.014 | -0.019 |
| | SD | 0.003 | 0.000 | 0.003 | 0.031 | 0.007 | 0.007 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | .4%HNO3 | 0.038 | 0.001 | 0.103 | 0.101 | 0.653 | 0.638 |
| | | 0.030 | 0.001 | 0.086 | 0.064 | 0.527 | 0.539 |
| | | 0.034 | 0.001 | 0.104 | 0.114 | 0.682 | 0.672 |
| | Mean | 0.034 | 0.001 | 0.098 | 0.093 | 0.620 | 0.616 |
| | SD | 0.004 | 0.000 | 0.010 | 0.026 | 0.082 | 0.069 |
| Inline filter | D.I. | 0.159 | 0.068 | 0.011 | 0.011 | -0.022 | -0.024 |
| | | 0.190 | 0.091 | 0.006 | 0.037 | -0.016 | -0.018 |
| | | 0.199 | 0.099 | 0.014 | 0.070 | -0.016 | -0.022 |
| | Mean | 0.183 | 0.086 | 0.010 | 0.039 | -0.018 | -0.021 |
| | SD | 0.021 | 0.016 | 0.004 | 0.030 | 0.004 | 0.003 |
| | R+D.I. | 0.010 | 0.001 | 0.014 | 0.035 | 0.004 | -0.001 |
| | | 0.008 | 0.001 | 0.007 | 0.002 | -0.021 | -0.026 |
| | | 0.010 | 0.003 | 0.004 | 0.010 | -0.022 | -0.027 |
| | Mean | 0.009 | 0.002 | 0.008 | 0.016 | -0.013 | -0.018 |
| | SD | 0.001 | 0.001 | 0.005 | 0.017 | 0.015 | 0.015 |
| | .4%HNO3 | 0.030 | 0.007 | 0.008 | 0.000 | -0.019 | -0.020 |
| | | 0.028 | 0.007 | 0.010 | -0.006 | -0.012 | -0.017 |
| | | 0.036 | 0.010 | 0.008 | 0.029 | -0.004 | -0.005 |
| | Mean | 0.031 | 0.008 | 0.009 | 0.008 | -0.012 | -0.014 |
| | SD | 0.004 | 0.002 | 0.001 | 0.019 | 0.008 | 0.008 |
| Inline filter | D.I. | 0.015 | 0.000 | 0.050 | 0.058 | 0.015 | 0.014 |
| | | 0.007 | 0.000 | 0.031 | 0.014 | 0.009 | 0.004 |
| | | 0.010 | -0.001 | 0.033 | 0.046 | 0.006 | 0.003 |
| | Mean | 0.011 | 0.000 | 0.038 | 0.039 | 0.010 | 0.007 |
| | SD | 0.004 | 0.000 | 0.010 | 0.023 | 0.005 | 0.006 |
| | R+D.I. | 0.001 | -0.001 | 0.004 | -0.025 | -0.020 | -0.022 |
| | | 0.000 | -0.001 | 0.002 | 0.002 | -0.019 | -0.022 |
| | | 0.001 | -0.001 | 0.008 | 0.003 | -0.016 | -0.015 |
| | Mean | 0.001 | -0.001 | 0.005 | -0.007 | -0.018 | -0.019 |
| | SD | 0.000 | 0.000 | 0.003 | 0.016 | 0.002 | 0.004 |
| **Type** | **Sample** | **Mn 55** | **Co 59** | **Ni 60** | **Ni 62** | **Cu 63** | **Cu 65** |
| | .4%HNO3 | 0.061 | 0.002 | 0.078 | 0.057 | 0.599 | 0.599 |
| | | 0.052 | 0.001 | 0.049 | 0.048 | 0.490 | 0.496 |
| | | 0.119 | 0.000 | 0.057 | 0.039 | 0.557 | 0.553 |
| | Mean | 0.077 | 0.001 | 0.062 | 0.048 | 0.549 | 0.549 |
| | SD | 0.036 | 0.001 | 0.015 | 0.009 | 0.055 | 0.052 |

| Type | Sample | Zn 66 | As 75 | Mo 95 | Mo 98 | Ag 107 | Ag 109 |
|---|---|---|---|---|---|---|---|
| Syringe filter | D.I. | -0.015 | 0.002 | 0.002 | 0.001 | 0.000 | 0.000 |
| | | -0.025 | -0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| | | -0.022 | 0.001 | 0.002 | 0.001 | 0.000 | 0.000 |
| | Mean | -0.021 | 0.001 | 0.002 | 0.001 | 0.000 | 0.000 |
| | SD | 0.005 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | 0.043 | 0.000 | 0.001 | 0.000 | 0.001 | 0.000 |
| | | 0.005 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.158 | 0.000 | 0.006 | 0.005 | 0.000 | 0.001 |
| | Mean | 0.069 | 0.000 | 0.003 | 0.002 | 0.000 | 0.000 |
| | SD | 0.080 | 0.000 | 0.003 | 0.003 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.746 | 0.010 | 0.007 | 0.008 | 0.000 | 0.000 |
| | | 0.571 | 0.008 | 0.007 | 0.008 | 0.000 | 0.000 |
| | Mean | 0.659 | 0.009 | 0.007 | 0.008 | 0.000 | 0.000 |
| | SD | 0.124 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | 113.221 | 0.006 | 0.003 | 0.003 | 0.000 | 0.000 |
| | | 125.688 | 0.007 | 0.002 | 0.003 | 0.000 | 0.000 |
| | Mean | 119.455 | 0.007 | 0.002 | 0.003 | 0.000 | 0.000 |
| | SD | 8.815 | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.033 | -0.002 | 0.000 | 0.001 | 0.000 | -0.001 |
| | | -0.007 | -0.003 | -0.001 | 0.000 | 0.000 | -0.001 |
| | | 0.019 | 0.000 | 0.002 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.015 | -0.002 | 0.000 | 0.001 | 0.000 | 0.000 |
| | SD | 0.020 | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | 0.008 | -0.001 | -0.001 | 0.000 | 0.000 | 0.000 |
| | | 0.006 | -0.003 | 0.000 | 0.001 | 0.000 | 0.000 |
| | | 0.182 | -0.001 | 0.000 | 0.001 | 0.000 | -0.001 |
| | Mean | 0.065 | -0.002 | 0.000 | 0.001 | 0.000 | -0.001 |
| | SD | 0.101 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.047 | -0.001 | 0.005 | 0.005 | 0.001 | 0.000 |
| | | 0.042 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | -0.007 | -0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.028 | -0.001 | 0.002 | 0.002 | 0.000 | 0.000 |
| | SD | 0.030 | 0.002 | 0.002 | 0.002 | 0.000 | 0.000 |
| | .4%HNO3 | 0.075 | 0.000 | 0.000 | 0.001 | 0.000 | 0.000 |
| | | 0.124 | -0.001 | 0.001 | 0.001 | -0.001 | -0.001 |
| | | 0.098 | -0.002 | 0.001 | 0.001 | -0.001 | 0.000 |
| | Mean | 0.099 | -0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.025 | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| **Type** | **Sample** | **Zn 66** | **As 75** | **Mo 95** | **Mo 98** | **Ag 107** | **Ag 109** |
| Syringe filter | D.I. | -0.021 | 0.000 | 0.000 | 0.001 | 0.000 | -0.001 |
| | | -0.020 | 0.000 | 0.006 | 0.000 | 0.000 | 0.000 |
| | | -0.022 | -0.002 | -0.001 | 0.001 | 0.000 | 0.000 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Mean | -0.021 | 0.000 | 0.002 | 0.000 | 0.000 | 0.000 |
| | SD | 0.001 | 0.001 | 0.004 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | 0.016 | -0.002 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.331 | -0.001 | -0.001 | 0.001 | -0.001 | 0.000 |
| | | 0.133 | 0.000 | 0.001 | 0.003 | 0.000 | 0.000 |
| | Mean | 0.160 | -0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | SD | 0.159 | 0.001 | 0.001 | 0.001 | 0.001 | 0.000 |
| Syringe filter | D.I. | 0.072 | 0.001 | 0.001 | 0.003 | 0.000 | 0.000 |
| | | -0.022 | 0.001 | 0.002 | 0.002 | 0.000 | 0.000 |
| | | -0.015 | -0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.012 | 0.000 | 0.001 | 0.002 | 0.000 | 0.000 |
| | SD | 0.052 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | 0.068 | 0.000 | 0.000 | 0.001 | -0.001 | 0.000 |
| | | 0.030 | 0.000 | 0.001 | 0.000 | 0.000 | -0.001 |
| | | 0.205 | 0.002 | 0.001 | 0.000 | -0.001 | -0.001 |
| | Mean | 0.101 | 0.001 | 0.001 | 0.000 | -0.001 | -0.001 |
| | SD | 0.092 | 0.002 | 0.000 | 0.001 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.094 | -0.002 | 0.001 | 0.001 | -0.001 | -0.001 |
| | | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 | -0.001 |
| | | 0.013 | -0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.036 | -0.001 | 0.000 | 0.001 | -0.001 | -0.001 |
| | SD | 0.051 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | -0.006 | 0.001 | 0.000 | 0.000 | 0.000 | -0.001 |
| | | 0.003 | 0.001 | 0.002 | 0.003 | 0.001 | 0.000 |
| | | 0.094 | 0.001 | 0.003 | 0.003 | 0.000 | 0.000 |
| | Mean | 0.030 | 0.001 | 0.002 | 0.002 | 0.000 | 0.000 |
| | SD | 0.055 | 0.000 | 0.002 | 0.001 | 0.000 | 0.000 |
| Vacuum filter | D.I. | 0.100 | 0.001 | 0.008 | 0.007 | 0.000 | 0.000 |
| | | 0.034 | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| | | -0.002 | 0.002 | 0.000 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.044 | 0.001 | 0.003 | 0.003 | 0.000 | 0.000 |
| | SD | 0.052 | 0.000 | 0.004 | 0.004 | 0.000 | 0.000 |
| | R+D.I. | 0.005 | 0.000 | 0.000 | 0.000 | 0.000 | -0.001 |
| | | 0.001 | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | -0.014 | 0.001 | -0.001 | 0.001 | 0.000 | -0.001 |
| | Mean | -0.003 | 0.001 | 0.000 | 0.000 | 0.000 | -0.001 |
| | SD | 0.010 | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| **Type** | **Sample** | **Zn 66** | **As 75** | **Mo 95** | **Mo 98** | **Ag 107** | **Ag 109** |
| | .4%HNO3 | 0.809 | 0.001 | 0.000 | 0.001 | 0.000 | -0.001 |
| | | 0.215 | 0.002 | 0.000 | 0.000 | 0.000 | -0.001 |
| | | 0.130 | 0.002 | 0.001 | 0.001 | 0.000 | -0.001 |
| | Mean | 0.385 | 0.002 | 0.000 | 0.001 | 0.000 | -0.001 |
| | SD | 0.370 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| Vacuum | D.I. | 1.424 | 0.004 | 0.003 | 0.002 | 0.000 | 0.000 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| filter | | 1.336 | 0.002 | 0.004 | 0.004 | 0.000 | 0.000 |
| | | 1.382 | 0.003 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 1.381 | 0.003 | 0.003 | 0.002 | 0.000 | 0.000 |
| | SD | 0.044 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | R+D.I. | 0.174 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | | 0.127 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.188 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.163 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | SD | 0.032 | 0.000 | 0.000 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | 0.416 | 0.001 | -0.001 | 0.000 | 0.000 | 0.000 |
| | | 0.597 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.520 | 0.000 | -0.001 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.511 | 0.000 | -0.001 | 0.000 | 0.000 | 0.000 |
| | SD | 0.091 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| Vacuum filter | D.I. | 0.206 | 0.001 | | -0.001 | -0.001 | |
| | | 0.042 | -0.003 | | 0.001 | -0.001 | |
| | | 0.101 | 0.002 | | 0.000 | -0.002 | |
| | Mean | 0.116 | 0.000 | | 0.000 | -0.001 | |
| | SD | 0.083 | 0.003 | | 0.001 | 0.001 | |
| | R+D.I. | 0.095 | -0.003 | | 0.001 | -0.002 | |
| | | 0.123 | -0.001 | | 0.002 | -0.001 | |
| | | 0.072 | 0.001 | | 0.002 | -0.001 | |
| | Mean | 0.097 | -0.001 | | 0.002 | -0.001 | |
| | SD | 0.026 | 0.002 | | 0.001 | 0.001 | |
| | .4%HNO3 | 1.014 | 0.000 | | 0.000 | -0.001 | |
| | | 0.986 | -0.008 | | 0.002 | -0.002 | |
| | | 1.426 | -0.005 | | 0.000 | -0.002 | |
| | Mean | 1.142 | -0.004 | | 0.001 | -0.002 | |
| | SD | 0.246 | 0.004 | | 0.001 | 0.001 | |
| | D.I. | 0.016 | 0.000 | 0.000 | 0.001 | 0.000 | 0.000 |
| | R+D.I. | 0.002 | -0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | 0.652 | -0.002 | -0.001 | 0.000 | 0.000 | 0.000 |

| **Type** | **Sample** | **Zn 66** | **As 75** | **Mo 95** | **Mo 98** | **Ag 107** | **Ag 109** |
|---|---|---|---|---|---|---|---|
| Inline filter | D.I. | 0.616 | 0.012 | 0.023 | 0.021 | 0.000 | 0.000 |
| | | 0.312 | 0.005 | 0.014 | 0.015 | 0.000 | 0.000 |
| | | 0.210 | 0.006 | 0.015 | 0.016 | 0.000 | 0.000 |
| | Mean | 0.379 | 0.008 | 0.017 | 0.018 | 0.000 | 0.000 |
| | SD | 0.211 | 0.004 | 0.005 | 0.003 | 0.000 | 0.000 |
| | R+D.I. | 0.021 | 0.000 | 0.000 | 0.002 | 0.000 | 0.000 |
| | | 0.003 | 0.000 | 0.000 | 0.002 | 0.000 | 0.000 |
| | | -0.004 | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.006 | 0.000 | 0.000 | 0.001 | 0.000 | 0.000 |
| | SD | 0.013 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | .4%HNO3 | 1.772 | 0.002 | 0.000 | 0.002 | 0.000 | 0.000 |
| | | 1.274 | 0.001 | 0.001 | 0.002 | 0.000 | 0.000 |
| | | 2.206 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 1.751 | 0.001 | 0.001 | 0.002 | 0.000 | 0.000 |
| | SD | 0.466 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| Inline filter | D.I. | 0.030 | 0.001 | 0.000 | 0.000 | 0.000 | -0.001 |
| | | 0.018 | 0.002 | -0.001 | 0.000 | 0.000 | -0.001 |
| | | -0.007 | 0.003 | 0.002 | 0.001 | 0.000 | -0.001 |
| | Mean | 0.014 | 0.002 | 0.000 | 0.001 | 0.000 | -0.001 |
| | SD | 0.019 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | R+D.I. | -0.009 | 0.003 | 0.002 | 0.004 | 0.000 | 0.000 |
| | | 0.004 | 0.003 | 0.002 | 0.003 | 0.000 | 0.000 |
| | | -0.032 | 0.002 | 0.000 | 0.003 | 0.000 | 0.000 |
| | Mean | -0.012 | 0.003 | 0.001 | 0.004 | 0.000 | 0.000 |
| | SD | 0.019 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | 0.023 | 0.002 | 0.000 | 0.001 | 0.000 | 0.000 |
| | | 0.029 | 0.003 | 0.001 | 0.000 | 0.000 | 0.000 |
| | | 0.106 | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.053 | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 |
| | SD | 0.046 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| Inline filter | D.I. | 0.110 | 0.006 | 0.009 | 0.010 | 0.000 | 0.000 |
| | | 0.113 | 0.003 | 0.007 | 0.008 | 0.000 | 0.000 |
| | | 0.114 | 0.006 | 0.008 | 0.010 | 0.000 | 0.000 |
| | Mean | 0.112 | 0.005 | 0.008 | 0.009 | 0.000 | 0.000 |
| | SD | 0.002 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | R+D.I. | -0.016 | 0.001 | -0.001 | 0.000 | 0.000 | -0.001 |
| | | -0.015 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | | -0.009 | 0.000 | 0.002 | 0.003 | 0.001 | 0.001 |
| | Mean | -0.014 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | SD | 0.003 | 0.001 | 0.002 | 0.001 | 0.001 | 0.001 |

| **Type** | **Sample** | **Zn 66** | **As 75** | **Mo 95** | **Mo 98** | **Ag 107** | **Ag 109** |
|---|---|---|---|---|---|---|---|
| | .4%HNO3 | 2.167 | 0.003 | 0.006 | 0.007 | 0.001 | 0.001 |
| | | 1.692 | 0.001 | 0.004 | 0.006 | 0.000 | 0.001 |
| | | 2.035 | 0.001 | 0.005 | 0.006 | 0.000 | 0.000 |
| | Mean | 1.965 | 0.002 | 0.005 | 0.006 | 0.001 | 0.001 |
| | SD | 0.245 | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 |

| Type | Sample | Cd 111 | Cd 114 | Sb 121 | Sb 123 | Tl 203 | Tl 205 |
|---|---|---|---|---|---|---|---|
| Syringe filter | D.I. | 0.001 | 0.002 | 0.001 | 0.000 | 0.001 | 0.000 |
| | | 0.003 | 0.001 | 0.000 | 0.001 | 0.001 | 0.000 |
| | | 0.001 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.001 | 0.001 | 0.001 | 0.001 | 0.000 |
| | SD | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | 0.003 | 0.002 | 0.002 | 0.002 | 0.001 | 0.000 |
| | | 0.000 | 0.001 | 0.002 | 0.002 | 0.000 | 0.000 |
| | | 0.002 | 0.002 | 0.001 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.001 | 0.002 | 0.001 | 0.000 | 0.000 |
| | SD | 0.002 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.001 | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.001 | 0.002 | 0.003 | 0.003 | 0.000 | 0.000 |
| | Mean | 0.001 | 0.002 | 0.002 | 0.001 | 0.000 | 0.000 |
| | SD | 0.000 | 0.000 | 0.002 | 0.002 | 0.000 | 0.000 |
| | .4%HNO3 | 0.020 | 0.019 | 0.004 | 0.005 | 0.006 | 0.006 |
| | | 0.021 | 0.020 | 0.005 | 0.004 | 0.006 | 0.007 |
| | Mean | 0.020 | 0.020 | 0.005 | 0.005 | 0.006 | 0.006 |
| | SD | 0.001 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.002 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.006 | 0.007 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | -0.001 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.003 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.004 | 0.003 | 0.000 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | 0.003 | 0.004 | 0.003 | 0.003 | 0.000 | 0.000 |
| | | 0.000 | 0.005 | 0.002 | 0.001 | 0.000 | 0.000 |
| | | 0.002 | 0.004 | 0.002 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.004 | 0.002 | 0.002 | 0.000 | 0.000 |
| | SD | 0.002 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| Syringe filter | D.I. | -0.001 | 0.000 | -0.001 | -0.002 | 0.000 | 0.000 |
| | | 0.000 | 0.001 | 0.000 | 0.000 | 0.000 | 0.004 |
| | | -0.001 | 0.001 | 0.003 | 0.002 | 0.000 | 0.000 |
| | Mean | -0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.001 |
| | SD | 0.001 | 0.000 | 0.002 | 0.002 | 0.000 | 0.003 |
| | .4%HNO3 | 0.001 | 0.001 | 0.002 | 0.004 | 0.000 | 0.000 |
| | | -0.001 | 0.002 | 0.002 | 0.003 | 0.000 | 0.000 |
| | | 0.001 | 0.002 | 0.002 | 0.003 | 0.000 | 0.000 |
| | Mean | 0.000 | 0.001 | 0.002 | 0.003 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| **Type** | **Sample** | **Cd 111** | **Cd 114** | **Sb 121** | **Sb 123** | **Tl 203** | **Tl 205** |
| Syringe filter | D.I. | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| | | 0.001 | 0.001 | -0.001 | 0.000 | 0.000 | 0.000 |
| | | -0.001 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Mean | 0.000 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | 0.001 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.004 | 0.004 | 0.002 | 0.001 | 0.000 | 0.000 |
| | | 0.003 | 0.002 | 0.000 | -0.001 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.003 | 0.001 | 0.000 | 0.000 | 0.000 |
| | SD | 0.002 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.002 | 0.003 | 0.000 | -0.001 | 0.000 | 0.000 |
| | | 0.001 | 0.002 | 0.002 | 0.002 | 0.000 | 0.000 |
| | | 0.000 | 0.002 | 0.001 | 0.002 | 0.000 | 0.000 |
| | Mean | 0.001 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.001 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | 0.000 | 0.005 | 0.004 | 0.003 | 0.000 | 0.000 |
| | | 0.002 | 0.007 | 0.003 | 0.004 | 0.000 | 0.000 |
| | | 0.003 | 0.009 | 0.004 | 0.004 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.007 | 0.004 | 0.004 | 0.000 | 0.000 |
| | SD | 0.002 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| Syringe filter | D.I. | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | -0.001 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.002 | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | SD | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | 0.001 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.000 | 0.000 | -0.001 | -0.001 | 0.000 | 0.000 |
| | | 0.003 | 0.003 | 0.000 | 0.000 | -0.001 | 0.000 |
| | Mean | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | SD | 0.001 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| Vacuum filter | D.I. | 0.001 | 0.000 | 0.003 | 0.002 | 0.000 | 0.000 |
| | | 0.001 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.000 | 0.000 | 0.000 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.001 | 0.000 | 0.001 | 0.002 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | R+D.I. | 0.000 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.001 | -0.001 | 0.001 | 0.000 | 0.000 | 0.000 |
| | | 0.001 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | SD | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| **Type** | **Sample** | **Cd 111** | **Cd 114** | **Sb 121** | **Sb 123** | **Tl 203** | **Tl 205** |
| | .4%HNO3 | 0.003 | 0.003 | 0.001 | 0.002 | 0.000 | 0.000 |
| | | 0.003 | 0.002 | 0.001 | 0.000 | 0.000 | 0.000 |
| | | 0.001 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.000 | 0.001 | 0.000 | 0.000 |
| Vacuum | D.I. | 0.003 | 0.002 | -0.001 | -0.001 | 0.000 | 0.000 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| filter | | 0.001 | 0.001 | 0.000 | -0.001 | 0.000 | 0.000 |
| | | 0.002 | 0.002 | 0.002 | 0.002 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.001 | 0.002 | 0.000 | 0.000 |
| | R+D.I. | 0.000 | 0.001 | 0.001 | 0.002 | 0.000 | 0.000 |
| | | -0.002 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.001 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.000 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.002 | 0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | .4%HNO3 | -0.001 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.002 | 0.000 | 0.001 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.001 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.000 | 0.001 | 0.000 | 0.000 |
| Vacuum filter | D.I. | | 0.005 | 0.003 | | | 0.000 |
| | | | 0.002 | 0.002 | | | 0.000 |
| | | | 0.002 | 0.001 | | | 0.000 |
| | Mean | | 0.003 | 0.002 | | | 0.000 |
| | SD | | 0.002 | 0.001 | | | 0.000 |
| | R+D.I. | | 0.003 | 0.001 | | | 0.000 |
| | | | -0.001 | 0.002 | | | 0.000 |
| | | | 0.002 | 0.001 | | | 0.000 |
| | Mean | | 0.001 | 0.001 | | | 0.000 |
| | SD | | 0.002 | 0.001 | | | 0.000 |
| | .4%HNO3 | | 0.005 | 0.003 | | | 0.000 |
| | | | 0.004 | 0.002 | | | 0.000 |
| | | | 0.004 | 0.001 | | | 0.000 |
| | Mean | | 0.004 | 0.002 | | | 0.000 |
| | SD | | 0.001 | 0.001 | | | 0.000 |
| | D.I. | 0.000 | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | R+D.I. | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| | .4%HNO3 | 0.011 | 0.012 | 0.000 | 0.000 | 0.000 | 0.000 |

| **Type** | **Sample** | **Cd 111** | **Cd 114** | **Sb 121** | **Sb 123** | **Tl 203** | **Tl 205** |
|---|---|---|---|---|---|---|---|
| Inline filter | D.I. | 0.005 | 0.004 | 0.005 | 0.004 | 0.000 | 0.000 |
| | | 0.004 | 0.002 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.002 | 0.002 | 0.003 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.003 | 0.003 | 0.003 | 0.002 | 0.000 | 0.000 |
| | SD | 0.002 | 0.002 | 0.002 | 0.002 | 0.000 | 0.000 |
| | R+D.I. | 0.001 | 0.001 | 0.002 | 0.002 | 0.000 | 0.000 |
| | | 0.000 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 | -0.001 |
| | Mean | 0.001 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | .4%HNO3 | 0.008 | 0.007 | 0.009 | 0.008 | 0.000 | 0.000 |
| | | 0.005 | 0.004 | 0.007 | 0.007 | 0.000 | 0.000 |
| | | 0.011 | 0.011 | 0.006 | 0.007 | 0.000 | -0.001 |
| | Mean | 0.008 | 0.007 | 0.007 | 0.007 | 0.000 | 0.000 |
| | SD | 0.003 | 0.003 | 0.001 | 0.001 | 0.000 | 0.000 |
| Inline filter | D.I. | 0.001 | 0.000 | 0.270 | 0.267 | -0.001 | -0.001 |
| | | 0.001 | 0.000 | 0.305 | 0.307 | 0.000 | -0.001 |
| | | 0.001 | -0.001 | 0.300 | 0.303 | 0.000 | -0.001 |
| | Mean | 0.001 | 0.000 | 0.292 | 0.292 | 0.000 | -0.001 |
| | SD | 0.000 | 0.000 | 0.019 | 0.022 | 0.000 | 0.000 |
| | R+D.I. | 0.000 | 0.001 | 0.035 | 0.037 | 0.000 | 0.000 |
| | | 0.000 | 0.000 | 0.027 | 0.026 | 0.000 | 0.000 |
| | | 0.001 | 0.000 | 0.040 | 0.043 | -0.001 | 0.000 |
| | Mean | 0.000 | 0.000 | 0.034 | 0.035 | 0.000 | 0.000 |
| | SD | 0.000 | 0.001 | 0.007 | 0.008 | 0.000 | 0.000 |
| | .4%HNO3 | 0.000 | 0.001 | 0.231 | 0.230 | 0.000 | 0.000 |
| | | 0.000 | 0.000 | 0.243 | 0.245 | -0.001 | 0.000 |
| | | 0.002 | -0.001 | 0.220 | 0.227 | 0.000 | 0.000 |
| | Mean | 0.001 | 0.000 | 0.231 | 0.234 | 0.000 | 0.000 |
| | SD | 0.001 | 0.001 | 0.011 | 0.010 | 0.000 | 0.000 |
| Inline filter | D.I. | 0.002 | 0.000 | 0.001 | 0.002 | 0.000 | 0.000 |
| | | 0.002 | 0.001 | 0.000 | 0.002 | 0.000 | 0.000 |
| | | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 | 0.000 |
| | Mean | 0.002 | 0.000 | 0.001 | 0.001 | 0.000 | 0.000 |
| | SD | 0.000 | 0.001 | 0.000 | 0.001 | 0.000 | 0.000 |
| | R+D.I. | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.002 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| | | 0.000 | -0.001 | 0.001 | 0.001 | 0.000 | 0.000 |
| | Mean | 0.000 | -0.001 | 0.000 | 0.000 | 0.000 | 0.000 |
| | SD | 0.001 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| **Type** | **Sample** | **Cd 111** | **Cd 114** | **Sb 121** | **Sb 123** | **Tl 203** | **Tl 205** |
| | .4%HNO3 | 0.007 | 0.006 | 0.005 | 0.004 | 0.001 | 0.000 |
| | | 0.016 | 0.016 | 0.004 | 0.005 | 0.000 | 0.000 |
| | | 0.008 | 0.009 | 0.004 | 0.003 | 0.000 | 0.000 |
| | Mean | 0.010 | 0.011 | 0.004 | 0.004 | 0.000 | 0.000 |
| | SD | 0.005 | 0.005 | 0.000 | 0.001 | 0.000 | 0.000 |

| Type | Sample | Pb 208 | Hg-ppt | Se-ppt |
|---|---|---|---|---|
| Syringe filter | D.I. | 0.000 | -1 | <1 |
| | | 0.000 | -1 | <1 |
| | | 0.000 | <1 | <1 |
| | Mean | 0.000 | | |
| | SD | 0.000 | | |
| | .4%HNO3 | 0.003 | <1 | <1 |
| | | 0.003 | <1 | 1 |
| | | 0.005 | <1 | <1 |
| | Mean | 0.004 | | |
| | SD | 0.001 | | |
| Syringe filter | D.I. | 0.001 | 1 | <1 |
| | | 0.001 | <1 | -1 |
| | Mean | 0.001 | | |
| | SD | 0.000 | | |
| | .4%HNO3 | 0.642 | 1 | -2 |
| | | 0.690 | <1 | -1 |
| | Mean | 0.666 | | |
| | SD | 0.033 | | |
| Syringe filter | D.I. | 0.003 | <1 | -1 |
| | | 0.001 | 2 | -1 |
| | | 0.003 | <1 | -1 |
| | Mean | 0.002 | | |
| | SD | 0.001 | | |
| | .4%HNO3 | 0.004 | 1 | -2 |
| | | 0.003 | <1 | 1 |
| | | 0.005 | <1 | <1 |
| | Mean | 0.004 | | |
| | SD | 0.001 | | |
| Syringe filter | D.I. | 0.001 | <1 | <1 |
| | | 0.001 | 1 | <1 |
| | | 0.000 | <1 | <1 |
| | Mean | 0.001 | | |
| | SD | 0.000 | | |
| | .4%HNO3 | 0.008 | 1 | <1 |
| | | 0.014 | 1 | <1 |
| | | 0.007 | <1 | <1 |
| | Mean | 0.010 | | |
| | SD | 0.004 | | |
| **Type** | **Sample** | **Pb 208** | **Hg-ppt** | **Se-ppt** |
| Syringe filter | D.I. | 0.000 | <1 | 1 |
| | | 0.009 | <1 | <1 |
| | | 0.000 | <1 | 1 |

| | | | | |
|---|---|---|---|---|
| | Mean | 0.003 | | |
| | SD | 0.005 | | |
| | .4%HNO3 | 0.003 | <1 | <1 |
| | | 0.014 | <1 | 1 |
| | | 0.005 | 1 | <1 |
| | Mean | 0.007 | | |
| | SD | 0.006 | | |
| Syringe filter | D.I. | 0.013 | 1 | <1 |
| | | 0.000 | <1 | <1 |
| | | -0.001 | <1 | -1 |
| | Mean | 0.004 | | |
| | SD | 0.008 | | |
| | .4%HNO3 | 0.002 | <1 | -2 |
| | | 0.002 | <1 | -1 |
| | | 0.004 | <1 | -2 |
| | Mean | 0.003 | | |
| | SD | 0.001 | | |
| Syringe filter | D.I. | 0.001 | <1 | 1 |
| | | 0.000 | 1 | 1 |
| | | 0.001 | 1 | <1 |
| | Mean | 0.001 | | |
| | SD | 0.000 | | |
| | .4%HNO3 | 0.002 | <1 | -1 |
| | | 0.003 | <1 | -1 |
| | | 0.012 | <1 | -2 |
| | Mean | 0.006 | | |
| | SD | 0.006 | | |
| Vacuum filter | D.I. | 0.009 | 1 | -1 |
| | | 0.001 | <1 | -2 |
| | | 0.000 | <1 | -2 |
| | Mean | 0.003 | | |
| | SD | 0.005 | | |
| | R+D.I. | 0.000 | <1 | -1 |
| | | 0.000 | -1 | 2 |
| | | 0.000 | <1 | 1 |
| | Mean | 0.000 | | |
| | SD | 0.000 | | |

| **Type** | **Sample** | **Pb 208** | **Hg-ppt** | **Se-ppt** |
|---|---|---|---|---|
| | .4%HNO3 | 0.016 | -1 | <1 |
| | | 0.016 | 1 | -1 |
| | | 0.014 | -1 | -1 |
| | Mean | 0.015 | | |
| | SD | 0.001 | | |
| Vacuum | D.I. | 0.005 | <1 | -1 |

| | | | | |
|---|---|---|---|---|
| filter | | 0.000 | 1 | -1 |
| | | 0.082 | <1 | <1 |
| | Mean | 0.029 | | |
| | SD | 0.046 | | |
| | R+D.I. | 0.005 | -1 | -1 |
| | | -0.001 | -1 | -1 |
| | | 0.000 | -1 | 4 |
| | Mean | 0.002 | | |
| | SD | 0.003 | | |
| | .4%HNO3 | 0.004 | <1 | 2 |
| | | 0.004 | <1 | 1 |
| | | 0.006 | <1 | 1 |
| | Mean | 0.005 | | |
| | SD | 0.001 | | |
| Vacuum filter | D.I. | 0.028 | <1 | -1 |
| | | -0.004 | -1 | <1 |
| | | 0.012 | <1 | -1 |
| | Mean | 0.012 | | |
| | SD | 0.016 | | |
| | R+D.I. | 0.031 | <1 | -1 |
| | | -0.006 | <1 | -1 |
| | | 0.026 | <1 | -2 |
| | Mean | 0.017 | | |
| | SD | 0.020 | | |
| | .4%HNO3 | 0.117 | <1 | 3 |
| | | 0.065 | <1 | 1 |
| | | 0.090 | <1 | 1 |
| | Mean | 0.091 | | |
| | SD | 0.026 | | |
| | D.I. | 0.005 | <1 | <1 |
| | R+D.I. | 0.008 | -1 | <1 |
| | .4%HNO3 | 0.206 | -1 | <1 |

| **Type** | **Sample** | **Pb 208** | **Hg-ppt** | **Se-ppt** |
|---|---|---|---|---|
| Inline filter | D.I. | 0.018 | <1 | <1 |
| | | 0.015 | <1 | <1 |
| | | 0.013 | <1 | -1 |
| | Mean | 0.016 | | |
| | SD | 0.003 | | |
| | R+D.I. | 0.000 | <1 | -1 |
| | | 0.000 | <1 | 2 |
| | | 0.000 | 1 | <1 |
| | Mean | 0.000 | | |
| | SD | 0.000 | | |

| | | | | |
|---|---|---|---|---|
| | .4%HNO3 | 0.194 | -1 | <1 |
| | | 0.108 | -1 | <1 |
| | | 0.193 | <1 | <1 |
| | Mean | 0.165 | | |
| | SD | 0.049 | | |
| Inline filter | D.I. | 0.004 | <1 | <1 |
| | | 0.004 | <1 | -1 |
| | | 0.004 | <1 | -2 |
| | Mean | 0.004 | | |
| | SD | 0.000 | | |
| | R+D.I. | 0.001 | <1 | -1 |
| | | 0.003 | <1 | <1 |
| | | 0.002 | <1 | 4 |
| | Mean | 0.002 | | |
| | SD | 0.001 | | |
| | .4%HNO3 | 0.038 | <1 | <1 |
| | | 0.064 | <1 | 1 |
| | | 0.072 | <1 | -1 |
| | Mean | 0.058 | | |
| | SD | 0.018 | | |
| Inline filter | D.I. | 0.000 | <1 | <1 |
| | | 0.001 | <1 | -1 |
| | | 0.000 | <1 | -1 |
| | Mean | 0.000 | | |
| | SD | 0.000 | | |
| | R+D.I. | 0.000 | -1 | -1 |
| | | 0.000 | -1 | <1 |
| | | 0.000 | <1 | <1 |
| | Mean | 0.000 | | |
| | SD | 0.000 | | |

| Type | Sample | Pb 208 | Hg-ppt | Se-ppt |
|---|---|---|---|---|
| | .4%HNO3 | 0.018 | -1 | 3 |
| | | 0.030 | -1 | 1 |
| | | 0.022 | -1 | <1 |
| | Mean | 0.023 | | |
| | SD | 0.006 | | |

|  | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Ni 62 | Cu 63 | Cu 65 | Zn 66 | As 75 | Mo 95 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ott River | 116.386 | 0.501 | 100.133 | 8.924 | 0.035 | 0.574 | 0.748 | 1.245 | 1.324 | 0.959 | 0.793 | 0.257 |
| Ott River | 109.342 | 0.496 | 105.938 | 8.825 | 0.035 | 0.584 | 0.721 | 1.225 | 1.253 | 0.957 | 0.808 | 0.259 |
| Ott River | 108.092 | 0.489 | 107.064 | 9.103 | 0.034 | 0.585 | 0.736 | 1.248 | 1.224 | 0.960 | 0.804 | 0.259 |
| Mean | 111.273 | 0.495 | 104.378 | 8.951 | 0.035 | 0.581 | 0.735 | 1.239 | 1.267 | 0.959 | 0.802 | 0.258 |
| SD | 4.472 | 0.006 | 3.719 | 0.141 | 0.001 | 0.006 | 0.014 | 0.013 | 0.051 | 0.002 | 0.008 | 0.001 |
| G-1-S | 54.178 | 0.356 | 64.910 | 3.043 | 0.015 | 0.503 | 0.652 | 1.157 | 1.175 | 0.540 | 0.758 | 0.257 |
| G-1-S | 59.116 | 0.372 | 61.616 | 3.054 | 0.017 | 0.511 | 0.696 | 1.200 | 1.239 | 0.571 | 0.757 | 0.258 |
| G-1-S | 55.056 | 0.366 | 68.468 | 3.081 | 0.016 | 0.531 | 0.703 | 1.190 | 1.155 | 0.533 | 0.783 | 0.257 |
| Mean | 56.117 | 0.365 | 64.998 | 3.059 | 0.016 | 0.515 | 0.684 | 1.182 | 1.190 | 0.548 | 0.766 | 0.257 |
| SD | 2.634 | 0.008 | 3.427 | 0.020 | 0.001 | 0.014 | 0.028 | 0.023 | 0.044 | 0.020 | 0.015 | 0.001 |
| G-3-S | 43.797 | 0.343 | 65.014 | 2.852 | 0.016 | 0.584 | 0.738 | 1.239 | 1.182 | 0.537 | 0.769 | 0.258 |
| G-3-S | 41.332 | 0.341 | 62.033 | 2.757 | 0.015 | 0.486 | 0.586 | 1.196 | 1.161 | 0.532 | 0.769 | 0.253 |
| G-3-S | 41.035 | 0.353 | 61.849 | 2.773 | 0.014 | 0.436 | 0.537 | 1.173 | 1.126 | 0.540 | 0.784 | 0.251 |
| Mean | 42.055 | 0.346 | 62.965 | 2.794 | 0.015 | 0.502 | 0.620 | 1.203 | 1.156 | 0.536 | 0.774 | 0.254 |
| SD | 1.516 | 0.006 | 1.777 | 0.051 | 0.001 | 0.075 | 0.105 | 0.034 | 0.028 | 0.004 | 0.009 | 0.004 |
| Ott River | 108.707 | 0.517 | 103.932 | 8.464 | 0.036 | 0.521 | 0.667 | 1.235 | 1.234 | 0.878 | 0.817 | 0.249 |
| Ott River | 108.009 | 0.444 | 105.367 | 8.369 | 0.035 | 0.597 | 0.753 | 1.221 | 1.211 | 0.880 | 0.793 | 0.253 |
| Ott River | 106.395 | 0.465 | 105.791 | 8.553 | 0.033 | 0.604 | 0.746 | 1.241 | 1.201 | 0.856 | 0.798 | 0.255 |
| M-1-S | 81.247 | 0.381 | 83.999 | 3.531 | 0.027 | 0.592 | 0.692 | 1.230 | 1.212 | 0.674 | 0.790 | 0.257 |
| M-1-S | 80.891 | 0.402 | 81.991 | 3.507 | 0.029 | 0.592 | 0.700 | 1.228 | 1.200 | 0.659 | 0.785 | 0.259 |
| M-1-S | 79.143 | 0.377 | 80.101 | 3.445 | 0.027 | 0.616 | 0.736 | 1.233 | 1.209 | 0.692 | 0.781 | 0.257 |
| Mean | 80.427 | 0.387 | 82.030 | 3.494 | 0.028 | 0.600 | 0.709 | 1.230 | 1.207 | 0.675 | 0.785 | 0.258 |
| SD | 1.126 | 0.013 | 1.949 | 0.044 | 0.001 | 0.014 | 0.023 | 0.003 | 0.006 | 0.017 | 0.005 | 0.001 |
| M-2-S | 78.734 | 0.375 | 82.104 | 3.465 | 0.021 | 0.627 | 0.709 | 1.164 | 1.167 | 0.677 | 0.790 | 0.256 |
| M-2-S | 80.480 | 0.362 | 71.735 | 3.413 | 0.023 | 0.520 | 0.672 | 1.220 | 1.218 | 0.654 | 0.773 | 0.248 |
| M-2-S | 80.085 | 0.360 | 76.519 | 3.460 | 0.023 | 0.543 | 0.693 | 1.225 | 1.203 | 0.680 | 0.771 | 0.259 |

|  | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Ni 62 | Cu 63 | Cu 65 | Zn 66 | As 75 | Mo 95 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Mean | 79.766 | 0.366 | 76.786 | 3.446 | 0.022 | 0.563 | 0.691 | 1.203 | 1.196 | 0.670 | 0.778 | 0.254 |
| SD | 0.916 | 0.008 | 5.190 | 0.029 | 0.001 | 0.056 | 0.019 | 0.034 | 0.026 | 0.014 | 0.010 | 0.006 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| M-4-S | 57.492 | 0.343 | 67.534 | 2.925 | 0.017 | 0.548 | 0.673 | 1.192 | 1.166 | 0.581 | 0.785 | 0.256 |
| M-4-S | 57.693 | 0.358 | 71.006 | 2.977 | 0.018 | 0.536 | 0.671 | 1.196 | 1.153 | 0.601 | 0.788 | 0.255 |
| M-4-S | 59.284 | 0.358 | 68.768 | 2.969 | 0.018 | 0.546 | 0.713 | 1.214 | 1.193 | 0.617 | 0.778 | 0.252 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| Mean | 58.156 | 0.353 | 69.103 | 2.957 | 0.018 | 0.543 | 0.686 | 1.201 | 1.171 | 0.600 | 0.784 | 0.254 |
| SD | 0.982 | 0.009 | 1.760 | 0.028 | 0.001 | 0.006 | 0.024 | 0.012 | 0.020 | 0.018 | 0.005 | 0.002 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| Ott River | 107.295 | 0.464 | 102.621 | 8.403 | 0.033 | 0.604 | 0.736 | 1.228 | 1.234 | 0.888 | 0.807 | 0.263 |
| Ott River | 105.102 | 0.449 | 104.090 | 8.559 | 0.033 | 0.592 | 0.749 | 1.263 | 1.214 | 0.895 | 0.824 | 0.262 |
| Ott River | 106.032 | 0.439 | 104.624 | 8.664 | 0.036 | 0.631 | 0.747 | 1.272 | 1.234 | 0.945 | 0.809 | 0.260 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| G-1-L | 38.353 | 0.354 | 52.813 | 2.637 | 0.014 | 0.474 | 0.612 | 1.377 | 1.348 | 1.153 | 0.753 | 0.253 |
| G-1-L | 34.408 | 0.348 | 44.946 | 2.474 | 0.014 | 0.472 | 0.563 | 1.478 | 1.453 | 1.299 | 0.757 | 0.243 |
| G-1-L | 37.777 | 0.389 | 54.771 | 2.861 | 0.015 | 0.562 | 0.661 | 1.476 | 1.460 | 1.317 | 0.844 | 0.263 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| Mean | 36.846 | 0.364 | 50.843 | 2.657 | 0.014 | 0.503 | 0.612 | 1.444 | 1.420 | 1.256 | 0.785 | 0.253 |
| SD | 2.131 | 0.022 | 5.200 | 0.194 | 0.001 | 0.051 | 0.049 | 0.058 | 0.063 | 0.090 | 0.051 | 0.010 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| G-2-L | 56.989 | 0.401 | 65.430 | 3.223 | 0.018 | 0.541 | 0.660 | 1.211 | 1.210 | 0.655 | 0.859 | 0.264 |
| G-2-L | 56.860 | 0.382 | 67.646 | 3.212 | 0.018 | 0.578 | 0.692 | 1.210 | 1.187 | 0.614 | 0.836 | 0.259 |
| G-2-L | 59.825 | 0.378 | 69.042 | 3.262 | 0.019 | 0.566 | 0.694 | 1.226 | 1.221 | 0.632 | 0.823 | 0.266 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| Mean | 57.891 | 0.387 | 67.373 | 3.232 | 0.018 | 0.562 | 0.682 | 1.216 | 1.206 | 0.634 | 0.839 | 0.263 |
| SD | 1.676 | 0.012 | 1.821 | 0.026 | 0.001 | 0.019 | 0.019 | 0.009 | 0.017 | 0.021 | 0.018 | 0.004 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| Ott River | 109.245 | 0.456 | 106.181 | 9.059 | 0.035 | 0.621 | 0.753 | 1.275 | 1.262 | 0.931 | 0.836 | 0.262 |
| Ott River | 107.688 | 0.424 | 105.424 | 9.144 | 0.035 | 0.622 | 0.730 | 1.276 | 1.249 | 0.961 | 0.835 | 0.267 |
| Ott River | 110.558 | 0.423 | 102.728 | 9.073 | 0.036 | 0.619 | 0.757 | 1.270 | 1.277 | 0.920 | 0.828 | 0.263 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
| G-3-L | 37.676 | 0.361 | 50.616 | 2.606 | 0.013 | 0.565 | 0.654 | 1.275 | 1.292 | 0.780 | 0.791 | 0.264 |
| G-3-L | 38.677 | 0.323 | 53.059 | 2.679 | 0.015 | 0.527 | 0.644 | 1.561 | 1.563 | 1.101 | 0.779 | 0.254 |
| G-3-L | 37.808 | 0.312 | 47.263 | 2.570 | 0.014 | 0.429 | 0.482 | 1.213 | 1.220 | 0.735 | 0.770 | 0.253 |

|  | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Ni 62 | Cu 63 | Cu 65 | Zn 66 | As 75 | Mo 95 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Mean | 38.054 | 0.332 | 50.313 | 2.618 | 0.014 | 0.507 | 0.593 | 1.350 | 1.358 | 0.872 | 0.780 | 0.257 |
| SD | 0.544 | 0.026 | 2.910 | 0.056 | 0.001 | 0.070 | 0.097 | 0.186 | 0.181 | 0.200 | 0.011 | 0.006 |
| M-1-V | 80.471 | 0.364 | 78.605 | 3.526 | 0.022 | 0.475 | 0.603 | 1.262 | 1.248 | 0.678 | 0.807 | 0.263 |
| M-1-V | 78.070 | 0.355 | 80.006 | 3.433 | 0.021 | 0.492 | 0.642 | 1.213 | 1.199 | 0.710 | 0.791 | 0.265 |
| M-1-V | 80.303 | 0.370 | 79.080 | 3.405 | 0.021 | 0.503 | 0.627 | 1.241 | 1.251 | 0.677 | 0.799 | 0.262 |
| Mean | 79.615 | 0.363 | 79.230 | 3.455 | 0.021 | 0.490 | 0.624 | 1.239 | 1.233 | 0.688 | 0.799 | 0.263 |
| SD | 1.340 | 0.008 | 0.712 | 0.063 | 0.001 | 0.014 | 0.020 | 0.025 | 0.029 | 0.019 | 0.008 | 0.002 |
| M-2-V | 65.031 | 0.353 | 74.606 | 3.093 | 0.019 | 0.532 | 0.642 | 1.233 | 1.208 | 0.709 | 0.796 | 0.265 |
| M-2-V | 67.730 | 0.359 | 75.974 | 3.123 | 0.020 | 0.565 | 0.684 | 1.254 | 1.239 | 0.670 | 0.807 | 0.257 |
| M-2-V | 68.805 | 0.385 | 77.845 | 3.146 | 0.021 | 0.584 | 0.750 | 1.254 | 1.270 | 0.759 | 0.811 | 0.269 |
| Mean | 67.189 | 0.366 | 76.142 | 3.121 | 0.020 | 0.560 | 0.692 | 1.247 | 1.239 | 0.713 | 0.805 | 0.264 |
| SD | 1.944 | 0.017 | 1.626 | 0.027 | 0.001 | 0.026 | 0.054 | 0.012 | 0.031 | 0.045 | 0.008 | 0.006 |
| Ott River | 112.729 | 0.431 | 112.923 | 8.716 | 0.036 | 0.646 | 0.818 | 1.284 | 1.296 | 1.023 | 0.820 | 0.264 |
| Ott River | 111.786 | 0.425 | 114.538 | 8.800 | 0.034 | 0.676 | 0.806 | 1.305 | 1.286 | 0.910 | 0.838 | 0.270 |
| Ott River | 112.343 | 0.419 | 113.215 | 8.694 | 0.036 | 0.611 | 0.745 | 1.274 | 1.314 | 0.897 | 0.824 | 0.266 |

|  | Mo 98 | Ag 107 | Ag 109 | Cd 111 | Cd 114 | Sb 121 | Sb 123 | Tl 203 | Tl 205 | Pb 208 | Se, ppt | Hg, ppt |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ott River | 0.260 | 0.003 | 0.002 | 0.016 | 0.015 | 0.071 | 0.071 | 0.007 | 0.008 | 0.150 | 84.0 | 56.0 |
| Ott River | 0.258 | 0.002 | 0.002 | 0.015 | 0.016 | 0.072 | 0.070 | 0.006 | 0.008 | 0.154 | 85.0 | 55.0 |
| Ott River | 0.252 | 0.002 | 0.002 | 0.014 | 0.014 | 0.068 | 0.069 | 0.006 | 0.007 | 0.152 | 85.0 | 55.0 |
| Mean | 0.257 | 0.002 | 0.002 | 0.015 | 0.015 | 0.070 | 0.070 | 0.006 | 0.008 | 0.152 | 84.7 | 55.3 |
| SD | 0.004 | 0.001 | 0.000 | 0.001 | 0.001 | 0.002 | 0.001 | 0.001 | 0.001 | 0.002 | 0.6 | 0.6 |
| G-1-S | 0.249 | 0.001 | 0.001 | 0.012 | 0.011 | 0.067 | 0.070 | 0.007 | 0.007 | 0.085 | 85.0 | 41.0 |
| G-1-S | 0.257 | 0.001 | 0.001 | 0.014 | 0.011 | 0.067 | 0.071 | 0.006 | 0.007 | 0.084 | 84.0 | 40.0 |
| G-1-S | 0.256 | 0.002 | 0.002 | 0.012 | 0.012 | 0.069 | 0.069 | 0.006 | 0.007 | 0.084 | 86.0 | 39.0 |
| Mean | 0.254 | 0.001 | 0.001 | 0.013 | 0.011 | 0.068 | 0.070 | 0.006 | 0.007 | 0.084 | 85.0 | 40.0 |
| SD | 0.004 | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 | 0.000 | 0.001 | 1.0 | 1.0 |
| G-3-S | 0.260 | 0.001 | 0.001 | 0.014 | 0.012 | 0.071 | 0.071 | 0.006 | 0.007 | 0.077 | 81.0 | 33.0 |
| G-3-S | 0.253 | 0.001 | 0.001 | 0.014 | 0.011 | 0.069 | 0.069 | 0.005 | 0.007 | 0.074 | 87.0 | 33.0 |
| G-3-S | 0.250 | 0.001 | 0.001 | 0.010 | 0.014 | 0.068 | 0.068 | 0.005 | 0.006 | 0.075 | 84.0 | 33.0 |
| Mean | 0.254 | 0.001 | 0.001 | 0.013 | 0.012 | 0.069 | 0.069 | 0.005 | 0.007 | 0.075 | 84.0 | 33.0 |
| SD | 0.005 | 0.000 | 0.000 | 0.002 | 0.002 | 0.002 | 0.002 | 0.001 | 0.001 | 0.002 | 3.0 | 0.0 |
| Ott River | 0.252 | 0.002 | 0.001 | 0.015 | 0.014 | 0.069 | 0.070 | 0.008 | 0.007 | 0.143 | 87.0 | 53.0 |
| Ott River | 0.254 | 0.002 | 0.003 | 0.013 | 0.015 | 0.072 | 0.070 | 0.007 | 0.007 | 0.145 | 87.0 | 55.0 |
| Ott River | 0.257 | 0.002 | 0.002 | 0.014 | 0.015 | 0.071 | 0.074 | 0.006 | 0.007 | 0.145 | 88.0 | 55.0 |
| M-1-S | 0.254 | 0.001 | 0.001 | 0.011 | 0.012 | 0.070 | 0.071 | 0.006 | 0.007 | 0.103 | 86.0 | 44.0 |
| M-1-S | 0.254 | 0.001 | 0.001 | 0.013 | 0.012 | 0.070 | 0.069 | 0.006 | 0.007 | 0.104 | 81.0 | 44.0 |
| M-1-S | 0.258 | 0.001 | 0.001 | 0.010 | 0.010 | 0.071 | 0.069 | 0.007 | 0.007 | 0.100 | 84.0 | 44.0 |
| Mean | 0.255 | 0.001 | 0.001 | 0.011 | 0.011 | 0.070 | 0.070 | 0.006 | 0.007 | 0.102 | 83.7 | 44.0 |
| SD | 0.002 | 0.000 | 0.000 | 0.002 | 0.001 | 0.001 | 0.001 | 0.001 | 0.000 | 0.002 | 2.5 | 0.0 |
| M-2-S | 0.258 | 0.001 | 0.001 | 0.012 | 0.012 | 0.068 | 0.069 | 0.006 | 0.007 | 0.100 | 84.0 | 43.0 |
| M-2-S | 0.244 | 0.003 | 0.002 | 0.012 | 0.010 | 0.071 | 0.072 | 0.007 | 0.007 | 0.098 | 84.0 | 42.0 |
| M-2-S | 0.257 | 0.002 | 0.003 | 0.015 | 0.013 | 0.072 | 0.073 | 0.008 | 0.008 | 0.101 | 81.0 | 42.0 |

|  | Mo 98 | Ag 107 | Ag 109 | Cd 111 | Cd 114 | Sb 121 | Sb 123 | Tl 203 | Tl 205 | Pb 208 | Se, ppt | Hg, ppt |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Mean | 0.253 | 0.002 | 0.002 | 0.013 | 0.012 | 0.070 | 0.071 | 0.007 | 0.007 | 0.100 | 83.0 | 42.3 |
| SD | 0.008 | 0.001 | 0.001 | 0.002 | 0.002 | 0.002 | 0.002 | 0.001 | 0.001 | 0.002 | 1.7 | 0.6 |
| M-4-S | 0.253 | 0.001 | 0.001 | 0.011 | 0.010 | 0.072 | 0.069 | 0.006 | 0.007 | 0.085 | 82.0 | 12.0 |
| M-4-S | 0.252 | 0.001 | 0.001 | 0.010 | 0.012 | 0.070 | 0.068 | 0.006 | 0.007 | 0.088 | 84.0 | 13.0 |
| M-4-S | 0.252 | 0.001 | 0.001 | 0.011 | 0.011 | 0.067 | 0.069 | 0.006 | 0.007 | 0.086 | 84.0 | 13.0 |
| Mean | 0.252 | 0.001 | 0.001 | 0.011 | 0.011 | 0.070 | 0.069 | 0.006 | 0.007 | 0.086 | 83.3 | 12.7 |
| SD | 0.001 | 0.000 | 0.000 | 0.001 | 0.001 | 0.003 | 0.001 | 0.000 | 0.000 | 0.002 | 1.2 | 0.6 |
| Ott River | 0.258 | 0.001 | 0.001 | 0.016 | 0.014 | 0.070 | 0.068 | 0.007 | 0.007 | 0.146 | 87.0 | 54.0 |
| Ott River | 0.255 | 0.002 | 0.002 | 0.014 | 0.016 | 0.072 | 0.074 | 0.006 | 0.008 | 0.148 | 81.0 | 55.0 |
| Ott River | 0.256 | 0.002 | 0.002 | 0.016 | 0.016 | 0.075 | 0.075 | 0.007 | 0.008 | 0.149 | 84.0 | 54.0 |
| G-1-L | 0.245 | 0.001 | 0.000 | 0.013 | 0.012 | 0.070 | 0.067 | 0.006 | 0.006 | 0.089 | 79.0 | 18.0 |
| G-1-L | 0.245 | 0.001 | 0.000 | 0.013 | 0.013 | 0.069 | 0.068 | 0.005 | 0.006 | 0.096 | 80.0 | 19.0 |
| G-1-L | 0.262 | 0.001 | 0.001 | 0.015 | 0.016 | 0.075 | 0.073 | 0.008 | 0.007 | 0.103 | 79.0 | 19.0 |
| Mean | 0.251 | 0.001 | 0.000 | 0.014 | 0.014 | 0.071 | 0.069 | 0.006 | 0.006 | 0.096 | 79.3 | 18.7 |
| SD | 0.010 | 0.000 | 0.001 | 0.001 | 0.002 | 0.003 | 0.003 | 0.002 | 0.001 | 0.007 | 0.6 | 0.6 |
| G-2-L | 0.259 | 0.002 | 0.001 | 0.010 | 0.014 | 0.097 | 0.097 | 0.006 | 0.007 | 0.098 | 80.0 | 34.0 |
| G-2-L | 0.267 | 0.003 | 0.002 | 0.011 | 0.012 | 0.085 | 0.083 | 0.006 | 0.008 | 0.095 | 79.0 | 34.0 |
| G-2-L | 0.268 | 0.002 | 0.002 | 0.013 | 0.014 | 0.091 | 0.090 | 0.007 | 0.007 | 0.099 | 82.0 | 35.0 |
| Mean | 0.265 | 0.002 | 0.002 | 0.011 | 0.013 | 0.091 | 0.090 | 0.006 | 0.007 | 0.097 | 80.3 | 34.3 |
| SD | 0.005 | 0.001 | 0.001 | 0.002 | 0.001 | 0.006 | 0.007 | 0.001 | 0.001 | 0.002 | 1.5 | 0.6 |
| Ott River | 0.265 | 0.002 | 0.002 | 0.014 | 0.016 | 0.072 | 0.073 | 0.007 | 0.007 | 0.154 | 85.0 | 53.0 |
| Ott River | 0.266 | 0.002 | 0.002 | 0.015 | 0.016 | 0.074 | 0.072 | 0.007 | 0.008 | 0.155 | 85.0 | 52.0 |
| Ott River | 0.267 | 0.002 | 0.002 | 0.016 | 0.016 | 0.072 | 0.072 | 0.006 | 0.007 | 0.153 | 88.0 | 54.0 |
| G-3-L | 0.265 | 0.001 | 0.000 | 0.012 | 0.012 | 0.069 | 0.069 | 0.006 | 0.007 | 0.066 | 82.0 | 20.0 |
| G-3-L | 0.260 | 0.002 | 0.001 | 0.013 | 0.013 | 0.072 | 0.071 | 0.005 | 0.007 | 0.077 | 80.0 | 18.0 |
| G-3-L | 0.252 | 0.001 | 0.001 | 0.012 | 0.012 | 0.072 | 0.072 | 0.006 | 0.006 | 0.064 | 81.0 | 20.0 |

|  | Mo 98 | Ag 107 | Ag 109 | Cd 111 | Cd 114 | Sb 121 | Sb 123 | Tl 203 | Tl 205 | Pb 208 | Se, ppt | Hg, ppt |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Mean | 0.259 | 0.001 | 0.001 | 0.012 | 0.012 | 0.071 | 0.071 | 0.006 | 0.007 | 0.069 | 81.0 | 19.3 |
| SD | 0.007 | 0.001 | 0.001 | 0.001 | 0.001 | 0.002 | 0.002 | 0.001 | 0.001 | 0.007 | 1.0 | 1.2 |
| M-1-V | 0.266 | 0.002 | 0.002 | 0.013 | 0.014 | 0.071 | 0.071 | 0.006 | 0.007 | 0.104 | 82.0 | 39.0 |
| M-1-V | 0.259 | 0.001 | 0.002 | 0.013 | 0.013 | 0.069 | 0.069 | 0.007 | 0.007 | 0.106 | 81.0 | 37.0 |
| M-1-V | 0.265 | 0.001 | 0.001 | 0.015 | 0.012 | 0.070 | 0.067 | 0.007 | 0.007 | 0.104 | 82.0 | 38.0 |
| Mean | 0.263 | 0.001 | 0.002 | 0.014 | 0.013 | 0.070 | 0.069 | 0.007 | 0.007 | 0.105 | 81.7 | 38.0 |
| SD | 0.004 | 0.001 | 0.001 | 0.001 | 0.001 | 0.001 | 0.002 | 0.001 | 0.000 | 0.001 | 0.6 | 1.0 |
| M-2-V | 0.264 | 0.001 | 0.001 | 0.017 | 0.017 | 0.072 | 0.070 | 0.005 | 0.006 | 0.100 | 86.0 | 32.0 |
| M-2-V | 0.263 | 0.003 | 0.002 | 0.012 | 0.013 | 0.077 | 0.077 | 0.005 | 0.005 | 0.094 | 87.0 | 34.0 |
| M-2-V | 0.267 | 0.002 | 0.002 | 0.018 | 0.015 | 0.073 | 0.073 | 0.006 | 0.006 | 0.103 | 84.0 | 33.0 |
| Mean | 0.265 | 0.002 | 0.002 | 0.016 | 0.015 | 0.074 | 0.073 | 0.005 | 0.006 | 0.099 | 85.7 | 33.0 |
| SD | 0.002 | 0.001 | 0.001 | 0.003 | 0.002 | 0.003 | 0.004 | 0.001 | 0.000 | 0.005 | 1.5 | 1.0 |
| Ott River | 0.272 | 0.002 | 0.002 | 0.016 | 0.015 | 0.074 | 0.074 | 0.008 | 0.008 | 0.156 | 85.0 | 54.0 |
| Ott River | 0.272 | 0.002 | 0.002 | 0.016 | 0.017 | 0.074 | 0.072 | 0.007 | 0.008 | 0.156 | 84.0 | 55.0 |
| Ott River | 0.263 | 0.002 | 0.002 | 0.017 | 0.015 | 0.070 | 0.071 | 0.008 | 0.007 | 0.152 | 86.0 | 53.0 |

## Filtration of spiked water samples at 0.45 um, Appendix 4.2
## Values in ppb except Se and Hg

| | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Cu 65 | Zn 66 |
|---|---|---|---|---|---|---|---|---|
| G-1-S | 24.8 | 9.9 | 49.9 | 9.8 | 9.8 | 9.9 | 9.8 | 9.9 |
| G-1-S | 25.0 | 10.1 | 48.9 | 10.0 | 9.9 | 10.0 | 9.9 | 10.1 |
| G-1-S | 24.6 | 10.0 | 49.2 | 10.1 | 10.0 | 10.0 | 10.0 | 10.0 |
| | 24.8 | 10.0 | 49.3 | 10.0 | 9.9 | 10.0 | 9.9 | 10.0 |
| | 0.2 | 0.1 | 0.5 | 0.2 | 0.1 | 0.1 | 0.1 | 0.1 |
| G-3-S | 24.8 | 10.0 | 48.3 | 9.8 | 10.0 | 10.1 | 10.0 | 10.1 |
| G-3-S | 25.1 | 10.2 | 48.3 | 10.0 | 10.2 | 10.2 | 10.3 | 10.2 |
| G-3-S | 25.4 | 10.3 | 49.5 | 10.1 | 10.2 | 10.3 | 10.3 | 10.3 |
| | 25.1 | 10.2 | 48.7 | 10.0 | 10.1 | 10.2 | 10.2 | 10.2 |
| | 0.3 | 0.2 | 0.7 | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 |
| M-1-S | 24.9 | 10.1 | 46.2 | 10.1 | 10.0 | 10.2 | 10.0 | 10.3 |
| M-1-S | 25.2 | 10.0 | 47.1 | 10.1 | 9.9 | 10.3 | 9.9 | 10.3 |
| M-1-S | 25.0 | 10.2 | 47.2 | 10.0 | 10.1 | 10.2 | 10.2 | 10.2 |
| | 25.0 | 10.1 | 46.8 | 10.1 | 10.0 | 10.2 | 10.0 | 10.3 |
| | 0.2 | 0.1 | 0.6 | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 |
| M-2-S | 25.5 | 10.1 | 48.8 | 10.1 | 10.1 | 10.2 | 10.1 | 10.2 |
| M-2-S | 25.6 | 10.2 | 50.1 | 10.1 | 10.1 | 10.1 | 10.0 | 10.2 |
| M-2-S | 25.9 | 10.3 | 49.0 | 9.9 | 10.1 | 10.3 | 10.1 | 10.3 |
| | 25.7 | 10.2 | 49.3 | 10.0 | 10.1 | 10.2 | 10.1 | 10.2 |
| | 0.2 | 0.1 | 0.7 | 0.1 | 0.0 | 0.1 | 0.1 | 0.1 |
| M-4-S | 25.5 | 10.1 | 49.2 | 10.0 | 10.1 | 10.2 | 10.0 | 10.1 |
| M-4-S | 24.8 | 10.0 | 51.0 | 10.0 | 10.1 | 10.1 | 10.0 | 10.0 |
| M-4-S | 25.0 | 10.0 | 49.7 | 9.9 | 10.2 | 10.1 | 10.2 | 10.3 |
| | 25.1 | 10.0 | 50.0 | 10.0 | 10.1 | 10.1 | 10.1 | 10.1 |
| | 0.4 | 0.1 | 0.9 | 0.1 | 0.1 | 0.1 | 0.1 | 0.2 |
| Whatman I | 25.4 | 10.1 | 48.0 | 9.9 | 10.1 | 10.1 | 10.1 | 10.1 |
| Whatman I | 24.6 | 9.9 | 50.3 | 9.9 | 9.9 | 9.9 | 9.9 | 9.8 |
| Whatman I | 24.7 | 10.0 | 51.8 | 10.1 | 10.1 | 10.0 | 10.2 | 10.0 |
| | 24.9 | 10.0 | 50.0 | 10.0 | 10.0 | 10.0 | 10.1 | 10.0 |
| | 0.4 | 0.1 | 1.9 | 0.1 | 0.1 | 0.1 | 0.2 | 0.2 |
| G-1-L | 25.3 | 9.9 | 48.1 | 9.8 | 10.0 | 10.1 | 10.3 | 10.8 |
| G-1-L | 24.5 | 9.9 | 50.7 | 10.0 | 10.0 | 10.0 | 10.3 | 10.3 |
| G-1-L | 24.7 | 9.8 | 49.4 | 9.9 | 10.0 | 9.9 | 10.1 | 10.2 |
| | 24.8 | 9.9 | 49.4 | 9.9 | 10.0 | 10.0 | 10.2 | 10.4 |
| | 0.4 | 0.1 | 1.3 | 0.1 | 0.0 | 0.1 | 0.1 | 0.3 |
| G-2-L | 24.4 | 9.9 | 51.8 | 10.1 | 10.0 | 10.0 | 10.0 | 9.9 |

## Filtration of spiked water samples at 0.45 um, Appendix 4.2
**Values in ppb except Se and Hg**

| | Al 27 | Cr 52 | Fe 54 | Mn 55 | Co 59 | Ni 60 | Cu 65 | Zn 66 |
|---|---|---|---|---|---|---|---|---|
| G-2-L | 24.5 | 10.0 | 52.0 | 10.0 | 10.1 | 10.0 | 10.1 | 9.8 |
| G-2-L | 24.7 | 9.9 | 51.5 | 10.0 | 9.9 | 9.9 | 9.8 | 9.9 |
| | 24.5 | 9.9 | 51.8 | 10.0 | 10.0 | 10.0 | 10.0 | 9.9 |
| | 0.2 | 0.1 | 0.3 | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 |
| G-3-L | 24.8 | 9.8 | 50.7 | 10.0 | 10.0 | 10.0 | 10.2 | 10.2 |
| G-3-L | 25.0 | 9.9 | 51.9 | 10.1 | 10.0 | 10.0 | 10.0 | 10.4 |
| G-3-L | 25.9 | 10.1 | 49.7 | 10.2 | 10.1 | 10.2 | 10.3 | 10.9 |
| | 25.2 | 9.9 | 50.8 | 10.1 | 10.0 | 10.1 | 10.2 | 10.5 |
| | 0.6 | 0.2 | 1.1 | 0.1 | 0.1 | 0.1 | 0.2 | 0.4 |
| M-1-V | 23.7 | 8.8 | 47.9 | 10.0 | 10.0 | 9.9 | 10.0 | 9.9 |
| M-1-V | 23.6 | 8.5 | 45.9 | 9.7 | 9.6 | 9.7 | 9.6 | 9.7 |
| M-1-V | 23.8 | 8.5 | 44.9 | 9.8 | 9.8 | 9.8 | 9.8 | 10.0 |
| | 23.7 | 8.6 | 46.2 | 9.8 | 9.8 | 9.8 | 9.8 | 9.9 |
| | 0.1 | 0.2 | 1.5 | 0.2 | 0.2 | 0.1 | 0.2 | 0.2 |
| M-2-V | 25.2 | 9.5 | 37.9 | 10.1 | 10.0 | 10.1 | 9.9 | 10.4 |
| M-2-V | 25.2 | 10.1 | 43.8 | 10.0 | 10.0 | 10.1 | 10.0 | 10.4 |
| M-2-V | 25.0 | 10.1 | 47.0 | 10.1 | 10.0 | 10.0 | 10.0 | 10.1 |
| | 25.1 | 9.9 | 42.9 | 10.1 | 10.0 | 10.1 | 10.0 | 10.3 |
| | 0.1 | 0.3 | 4.6 | 0.1 | 0.0 | 0.1 | 0.1 | 0.2 |

## Filtration of spiked water samples at 0.45 um, Appendix 4.2
## Values in ppb except Se and Hg

| | As 75 | Mo 98 | Ag 109 | Cd 111 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|
| G-1-S | 9.9 | 9.9 | 9.9 | 9.9 | 9.9 | 9.9 | 9.9 | 9.9 |
| G-1-S | 10.0 | 9.9 | 10.1 | 10.0 | 10.0 | 10.1 | 10.0 | 10.0 |
| G-1-S | 10.0 | 10.0 | 9.9 | 9.9 | 9.8 | 9.8 | 9.9 | 9.9 |
| | 10.0 | 9.9 | 10.0 | 9.9 | 9.9 | 9.9 | 9.9 | 9.9 |
| | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 |
| G-3-S | 9.9 | 9.9 | 10.0 | 9.9 | 9.9 | 9.7 | 10.0 | 10.0 |
| G-3-S | 10.0 | 10.1 | 10.0 | 9.9 | 10.0 | 9.8 | 10.0 | 10.0 |
| G-3-S | 10.2 | 10.1 | 10.1 | 10.0 | 10.0 | 9.9 | 10.0 | 10.0 |
| | 10.0 | 10.0 | 10.0 | 9.9 | 10.0 | 9.8 | 10.0 | 10.0 |
| | 0.2 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.0 | 0.0 |
| M-1-S | 10.0 | 10.0 | 10.0 | 9.9 | 10.0 | 9.9 | 9.9 | 10.0 |
| M-1-S | 10.0 | 10.0 | 10.1 | 10.1 | 10.2 | 10.3 | 10.0 | 10.2 |
| M-1-S | 10.0 | 10.0 | 10.0 | 10.0 | 10.1 | 9.8 | 10.0 | 10.1 |
| | 10.0 | 10.0 | 10.0 | 10.0 | 10.1 | 10.0 | 10.0 | 10.1 |
| | 0.0 | 0.0 | 0.1 | 0.1 | 0.1 | 0.3 | 0.1 | 0.1 |
| M-2-S | 10.1 | 10.1 | 10.1 | 10.1 | 10.0 | 10.0 | 10.0 | 10.0 |
| M-2-S | 10.0 | 10.1 | 10.1 | 10.1 | 10.1 | 10.1 | 10.0 | 10.0 |
| M-2-S | 10.0 | 9.9 | 10.1 | 9.9 | 10.1 | 9.9 | 10.0 | 10.0 |
| | 10.0 | 10.0 | 10.1 | 10.0 | 10.1 | 10.0 | 10.0 | 10.0 |
| | 0.1 | 0.1 | 0.0 | 0.1 | 0.1 | 0.1 | 0.0 | 0.0 |
| M-4-S | 9.9 | 10.0 | 10.1 | 10.0 | 10.0 | 10.1 | 10.0 | 10.0 |
| M-4-S | 9.9 | 10.0 | 10.0 | 9.9 | 9.9 | 9.5 | 10.0 | 10.0 |
| M-4-S | 10.0 | 9.9 | 10.0 | 9.9 | 10.0 | 9.4 | 10.0 | 10.0 |
| | 9.9 | 10.0 | 10.0 | 9.9 | 10.0 | 9.7 | 10.0 | 10.0 |
| | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.4 | 0.0 | 0.0 |
| Whatman I | 9.9 | 9.8 | 10.1 | 9.9 | 10.0 | 9.6 | 10.1 | 10.0 |
| Whatman I | 9.8 | 9.9 | 9.8 | 9.9 | 9.8 | 9.7 | 9.9 | 9.9 |
| Whatman I | 10.0 | 10.1 | 10.0 | 10.1 | 10.0 | 9.6 | 10.0 | 10.0 |
| | 9.9 | 9.9 | 10.0 | 10.0 | 9.9 | 9.6 | 10.0 | 10.0 |
| | 0.1 | 0.2 | 0.2 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| G-1-L | 9.8 | 9.8 | 10.0 | 9.9 | 9.9 | 9.8 | 10.0 | 9.9 |
| G-1-L | 9.9 | 9.9 | 10.0 | 10.0 | 9.9 | 9.7 | 9.9 | 9.9 |
| G-1-L | 9.8 | 9.9 | 9.9 | 9.9 | 9.8 | 9.8 | 9.9 | 9.9 |
| | 9.8 | 9.9 | 10.0 | 9.9 | 9.9 | 9.8 | 9.9 | 9.9 |
| | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.0 |
| G-2-L | 9.9 | 10.0 | 9.9 | 9.9 | 9.8 | 9.9 | 9.9 | 9.9 |

## Filtration of spiked water samples at 0.45 um, Appendix 4.2
## Values in ppb except Se and Hg

| | As 75 | Mo 98 | Ag 109 | Cd 111 | Cd 114 | Sb 121 | Tl 205 | Pb 208 |
|---|---|---|---|---|---|---|---|---|
| G-2-L | 10.0 | 10.0 | 9.9 | 10.0 | 9.8 | 9.6 | 9.9 | 10.0 |
| G-2-L | 9.9 | 10.0 | 9.9 | 9.9 | 9.9 | 10.3 | 10.0 | 10.1 |
| | 9.9 | 10.0 | 9.9 | 9.9 | 9.8 | 9.9 | 9.9 | 10.0 |
| | 0.1 | 0.0 | 0.0 | 0.1 | 0.1 | 0.4 | 0.1 | 0.1 |
| G-3-L | 10.1 | 10.1 | 9.9 | 9.9 | 9.9 | 10.0 | 9.9 | 10.0 |
| G-3-L | 10.2 | 10.2 | 9.9 | 10.0 | 10.1 | 10.5 | 10.1 | 10.2 |
| G-3-L | 10.1 | 10.2 | 10.0 | 10.1 | 10.3 | 10.4 | 10.2 | 10.2 |
| | 10.1 | 10.2 | 9.9 | 10.0 | 10.1 | 10.3 | 10.1 | 10.1 |
| | 0.1 | 0.1 | 0.1 | 0.1 | 0.2 | 0.3 | 0.2 | 0.1 |
| M-1-V | 9.9 | 10.2 | 10.0 | 9.9 | 9.9 | 10.1 | 10.0 | 9.6 |
| M-1-V | 9.7 | 9.8 | 9.8 | 9.7 | 9.7 | 10.0 | 9.7 | 9.3 |
| M-1-V | 9.7 | 9.8 | 9.9 | 9.8 | 9.9 | 9.7 | 9.7 | 9.4 |
| | 9.8 | 9.9 | 9.9 | 9.8 | 9.8 | 9.9 | 9.8 | 9.4 |
| | 0.1 | 0.2 | 0.1 | 0.1 | 0.1 | 0.2 | 0.2 | 0.2 |
| M-2-V | 10.0 | 9.9 | 9.2 | 10.0 | 10.0 | 9.6 | 7.5 | 9.0 |
| M-2-V | 10.0 | 9.9 | 9.5 | 9.9 | 9.9 | 9.7 | 8.4 | 9.6 |
| M-2-V | 10.0 | 10.0 | 9.6 | 10.1 | 10.0 | 9.7 | 8.8 | 9.9 |
| | 10.0 | 9.9 | 9.4 | 10.0 | 10.0 | 9.7 | 8.2 | 9.5 |
| | 0.0 | 0.1 | 0.2 | 0.1 | 0.1 | 0.1 | 0.7 | 0.5 |

## Filtration of spiked water samples at 0.45 um, Appendix 4.2
## Values in ppb except Se and Hg

| | Se, ppt | Hg, ppt | Hg, spike |
|---|---|---|---|
| G-1-S | 115.0 | 30.0 | 37.0 |
| G-1-S | 116.0 | 31.0 | |
| G-1-S | 120.0 | 30.0 | |
| | 117.0 | 30.3 | |
| | 2.6 | 0.6 | |
| G-3-S | 115.0 | 29.0 | |
| G-3-S | 115.0 | 28.0 | |
| G-3-S | 116.0 | 30.0 | |
| | | | 36.0 |
| | 115.3 | 29.0 | |
| | 0.6 | 1.0 | |
| M-1-S | 117.0 | 30.0 | |
| M-1-S | 119.0 | 31.0 | |
| M-1-S | 122.0 | 30.0 | |
| | 119.3 | 30.3 | |
| | 2.5 | 0.6 | |
| M-2-S | 117.0 | 30.0 | |
| M-2-S | 123.0 | 31.0 | |
| M-2-S | 114.0 | 32.0 | |
| | | | 35.0 |
| | 118.0 | 31.0 | |
| | 4.6 | 1.0 | |
| M-4-S | 115.0 | 27.0 | |
| M-4-S | 115.0 | 27.0 | |
| M-4-S | 114.0 | 27.0 | |
| | 114.7 | 27.0 | |
| | 0.6 | 0.0 | |
| Whatman I | 114.0 | 27.0 | |
| Whatman I | 113.0 | 27.0 | |
| Whatman I | 116.0 | 28.0 | |
| | | | 33.0 |
| | 114.3 | 27.3 | |
| | 1.5 | 0.6 | |
| G-1-L | 110.0 | 19.0 | |
| G-1-L | 111.0 | 22.0 | |
| G-1-L | 114.0 | 23.0 | |
| | 111.7 | 21.3 | |
| | 2.1 | 2.1 | |
| G-2-L | 112.0 | 35.0 | |

## Filtration of spiked water samples at 0.45 um, Appendix 4.2
## Values in ppb except Se and Hg

| | Se, ppt | Hg, ppt | Hg, spike |
|---|---|---|---|
| G-2-L | 115.0 | 25.0 | |
| G-2-L | 115.0 | 25.0 | |
| | | | 28.0 |
| | 114.0 | 28.3 | |
| | 1.7 | 5.8 | |
| G-3-L | 117.0 | 20.0 | |
| G-3-L | 114.0 | 20.0 | |
| G-3-L | 114.0 | 19.0 | |
| | 115.0 | 19.7 | |
| | 1.7 | 0.6 | |
| M-1-V | 111.0 | 23.0 | |
| M-1-V | 118.0 | 24.0 | |
| M-1-V | 117.0 | 23.0 | |
| | | | 25.0 |
| | 115.3 | 23.3 | |
| | 3.8 | 0.6 | |
| M-2-V | 114.0 | 18.0 | |
| M-2-V | 123.0 | 19.0 | |
| M-2-V | 113.0 | 18.0 | |
| | | | 24.0 |
| | 116.7 | 18.3 | |
| | 5.5 | 0.6 | |

# Stability study - Appendix 5.1

| | Day 0 | Day 1 | Day 7 | Day 14 | Day 21 | Day 28 |
|---|---|---|---|---|---|---|
| | **Al** | | | | | |
| SLRS-3 | 34.2 ± 0.6 | 33.0 ± 0.8 | 33.0 ± 0.3 | 33.9 ± 1.1 | 33.9 ± 0.5 | 33.5 ± 0.8 |
| OTT HDPE | 105.6 ± 2.5 | 98.1 ± 1.5 | 88.6 ± 1.1 | 96.5 ± 1.4 | 102.2 ± 4.5 | 95.5 ± 2.7 |
| OTT FEP | 106.3 ± 0.1 | 102.2 ± 1.1 | 90.2 ± 2.7 | 91.7 ± 0.8 | 100.0 ± 3.0 | 97.3 ± 0.9 |
| OTT PP | 104.6 ± 1.8 | 105.1 ± 4.1 | 91.4 ± 0.3 | 94.9 ± 1.7 | 104.1 ± 3.1 | 96.6 ± 3.6 |
| OTT PETG | 104.0 ± 1.3 | 102.9 ± 3.4 | 90.9 ± 0.5 | 100.1 ± 1.3 | 102.4 ± 0.2 | 92.0 ± 2.7 |
| RID HDPE | 7.0 ± 0.1 | 7.4 ± 0.1 | 7.1 ± 0.0 | 5.9 ± 0.1 | 7.3 ± 0.2 | 6.8 ± 0.1 |
| RID FEP | 6.7 ± 0.0 | 7.2 ± 0.0 | 7.0 ± 0.1 | 5.4 ± 0.2 | 7.2 ± 0.1 | 6.7 ± 0.2 |
| RID PP | 7.0 ± 0.0 | 6.9 ± 0.1 | 7.0 ± 0.0 | 5.5 ± 0.1 | 7.1 ± 0.1 | 7.1 ± 0.8 |
| RID PETG | 7.1 ± 0.1 | 6.9 ± 0.2 | 7.0 ± 0.0 | 5.9 ± 0.2 | 7.3 ± 0.2 | 6.4 ± 0.1 |
| GAT HDPE | 86.0 ± 0.5 | 79.5 ± 1.0 | 88.3 ± 2.1 | 85.2 ± 2.0 | 83.1 ± 0.3 | 85.5 ± 0.7 |
| GAT FEP | 85.6 ± 0.3 | 80.1 ± 0.6 | 85.4 ± 1.0 | 82.2 ± 1.1 | 83.4 ± 0.4 | 82.9 ± 2.3 |
| GAT PP | 84.3 ± 0.5 | 81.7 ± 0.3 | 83.7 ± 1.9 | 81.8 ± 1.6 | 85.0 ± 0.3 | 88.5 ± 1.0 |
| GAT PETG | 83.0 ± 1.3 | 80.8 ± 0.1 | 81.8 ± 0.4 | 79.6 ± 2.2 | 84.9 ± 0.3 | 85.8 ± 1.5 |
| SYN HDPE | 43.6 ± 0.1 | 41.6 ± 0.3 | 43.0 ± 0.8 | 45.5 ± 1.6 | 42.4 ± 0.3 | 44.2 ± 0.5 |
| SYN FEP | 43.4 ± 0.2 | 42.2 ± 0.3 | 43.4 ± 0.9 | 45.3 ± 0.3 | 42.6 ± 0.1 | 44.6 ± 0.1 |
| SYN PP | 43.5 ± 0.2 | 42.8 ± 0.3 | 43.4 ± 0.1 | 45.4 ± 0.4 | 42.7 ± 0.4 | 44.7 ± 0.2 |
| SYN PETG | 43.6 ± 0.1 | 43.1 ± 0.2 | 43.0 ± 0.6 | 45.4 ± 0.3 | 42.7 ± 0.3 | 44.4 ± 0.1 |
| | **Cr** | | | | | |
| SLRS-3 | 0.32 ± 0.01 | 0.29 ± 0.01 | 0.26 ± 0.01 | 0.31 ± 0.02 | 0.30 ± 0.01 | 0.30 ± 0.01 |
| OTT HDPE | 0.49 ± 0.02 | 0.40 ± 0.01 | 0.31 ± 0.00 | 0.33 ± 0.00 | 0.37 ± 0.01 | 0.33 ± 0.00 |
| OTT FEP | 0.50 ± 0.00 | 0.41 ± 0.01 | 0.29 ± 0.00 | 0.32 ± 0.01 | 0.34 ± 0.00 | 0.33 ± 0.01 |
| OTT PP | 0.48 ± 0.01 | 0.43 ± 0.02 | 0.30 ± 0.01 | 0.35 ± 0.00 | 0.36 ± 0.01 | 0.33 ± 0.01 |
| OTT PETG | 0.46 ± 0.01 | 0.42 ± 0.02 | 0.31 ± 0.01 | 0.35 ± 0.01 | 0.36 ± 0.01 | 0.32 ± 0.00 |
| RID HDPE | 1.21 ± 0.13 | 0.99 ± 0.18 | 0.27 ± 0.01 | 0.12 ± 0.01 | 0.15 ± 0.01 | 0.10 ± 0.00 |
| RID FEP | 1.32 ± 0.05 | 0.85 ± 0.15 | 0.18 ± 0.02 | 0.07 ± 0.01 | 0.11 ± 0.01 | 0.09 ± 0.00 |
| RID PP | 1.12 ± 0.01 | 0.89 ± 0.08 | 0.21 ± 0.02 | 0.10 ± 0.01 | 0.13 ± 0.01 | 0.09 ± 0.00 |
| RID PETG | 0.96 ± 0.03 | 0.91 ± 0.16 | 0.25 ± 0.01 | 0.10 ± 0.01 | 0.13 ± 0.00 | 0.07 ± 0.00 |
| GAT HDPE | 0.29 ± 0.01 | 0.22 ± 0.00 | 0.22 ± 0.01 | 0.23 ± 0.01 | 0.23 ± 0.00 | 0.24 ± 0.00 |
| GAT FEP | 0.28 ± 0.01 | 0.22 ± 0.01 | 0.20 ± 0.00 | 0.22 ± 0.01 | 0.22 ± 0.00 | 0.22 ± 0.01 |
| GAT PP | 0.27 ± 0.00 | 0.24 ± 0.00 | 0.20 ± 0.01 | 0.23 ± 0.01 | 0.24 ± 0.00 | 0.24 ± 0.00 |
| GAT PETG | 0.26 ± 0.01 | 0.24 ± 0.00 | 0.21 ± 0.01 | 0.21 ± 0.01 | 0.24 ± 0.00 | 0.23 ± 0.00 |
| SYN HDPE | 9.69 ± 0.07 | 9.25 ± 0.02 | 9.69 ± 0.08 | 9.79 ± 0.16 | 9.38 ± 0.08 | 9.85 ± 0.05 |
| SYN FEP | 9.61 ± 0.06 | 9.42 ± 0.10 | 9.59 ± 0.11 | 9.69 ± 0.09 | 9.47 ± 0.05 | 9.90 ± 0.09 |
| SYN PP | 9.68 ± 0.04 | 9.46 ± 0.13 | 9.62 ± 0.03 | 9.83 ± 0.26 | 9.47 ± 0.04 | 9.87 ± 0.04 |
| SYN PETG | 9.67 ± 0.07 | 9.44 ± 0.08 | 9.56 ± 0.05 | 9.89 ± 0.09 | 9.49 ± 0.07 | 9.83 ± 0.08 |
| | **Fe** | | | | | |
| SLRS-3 | 93.5 ± 1.2 | 88.9 ± 1.7 | 88.0 ± 2.6 | 89.4 ± 3.2 | 91.7 ± 4.1 | 91.7 ± 1.8 |
| OTT HDPE | 113.2 ± 3.0 | 105.1 ± 1.0 | 99.6 ± 0.8 | 109.6 ± 1.0 | 105.6 ± 3.2 | 111.0 ± 0.4 |
| OTT FEP | 117.4 ± 0.7 | 109.4 ± 2.7 | 100.5 ± 1.0 | 108.9 ± 1.3 | 104.2 ± 0.5 | 111.0 ± 1.7 |
| OTT PP | 114.9 ± 1.8 | 111.8 ± 4.6 | 101.0 ± 1.2 | 112.6 ± 2.2 | 107.0 ± 1.2 | 109.9 ± 4.0 |
| OTT PETG | 115.7 ± 1.2 | 107.6 ± 4.2 | 98.9 ± 1.2 | 114.0 ± 1.6 | 104.7 ± 1.0 | 103.5 ± 1.2 |
| RID HDPE | 19.1 ± 0.4 | 20.5 ± 0.6 | 20.7 ± 0.6 | 14.9 ± 1.1 | 16.8 ± 1.5 | 19.5 ± 0.2 |
| RID FEP | 18.6 ± 0.6 | 20.4 ± 1.0 | 20.7 ± 0.3 | 14.4 ± 0.3 | 17.7 ± 0.7 | 18.2 ± 0.2 |
| RID PP | 20.9 ± 0.2 | 19.3 ± 0.2 | 20.8 ± 0.4 | 15.3 ± 0.8 | 15.2 ± 0.2 | 17.7 ± 0.2 |
| RID PETG | 21.8 ± 0.1 | 18.9 ± 0.5 | 20.1 ± 0.2 | 14.7 ± 0.5 | 15.4 ± 0.5 | 16.4 ± 0.5 |
| GAT HDPE | 87.8 ± 0.6 | 78.2 ± 0.9 | 91.0 ± 2.0 | 86.9 ± 1.7 | 79.5 ± 0.6 | 89.5 ± 0.6 |
| GAT FEP | 87.4 ± 1.0 | 81.6 ± 1.7 | 86.4 ± 0.5 | 83.9 ± 0.4 | 80.0 ± 0.5 | 87.1 ± 1.9 |
| GAT PP | 85.2 ± 0.7 | 84.5 ± 1.8 | 86.1 ± 3.0 | 84.1 ± 0.5 | 84.0 ± 1.2 | 92.2 ± 1.4 |
| GAT PETG | 84.6 ± 1.7 | 82.9 ± 1.1 | 84.9 ± 1.5 | 82.7 ± 0.7 | 83.2 ± 0.6 | 89.5 ± 1.5 |
| SYN HDPE | 86.7 ± 1.0 | 81.4 ± 1.0 | 88.8 ± 0.8 | 89.9 ± 2.3 | 86.6 ± 1.3 | 90.5 ± 0.1 |
| SYN FEP | 85.1 ± 0.2 | 83.7 ± 0.7 | 88.9 ± 1.7 | 88.0 ± 0.1 | 86.3 ± 0.7 | 91.6 ± 1.0 |
| SYN PP | 87.7 ± 0.3 | 83.2 ± 0.9 | 89.1 ± 0.7 | 89.3 ± 1.0 | 87.2 ± 1.5 | 91.5 ± 0.6 |
| SYN PETG | 88.3 ± 1.0 | 81.6 ± 1.4 | 87.8 ± 0.7 | 87.6 ± 0.5 | 88.2 ± 1.2 | 90.8 ± 0.4 |

## Stability study - Appendix 5.1

| | Day 0 | Day 1 | Day 7 | Day 14 | Day 21 | Day 28 |
|---|---|---|---|---|---|---|
| | **Cu** | | | | | |
| SLRS-3 | 1.559 ± 0.015 | 1.540 ± 0.019 | 1.507 ± 0.042 | 1.628 ± 0.039 | 1.586 ± 0.030 | 1.562 ± 0.008 |
| OTT HDPE | 1.077 ± 0.025 | 1.052 ± 0.009 | 0.973 ± 0.013 | 1.104 ± 0.013 | 1.184 ± 0.035 | 1.048 ± 0.012 |
| OTT FEP | 1.129 ± 0.021 | 1.085 ± 0.031 | 0.990 ± 0.007 | 1.092 ± 0.010 | 1.161 ± 0.029 | 1.039 ± 0.005 |
| OTT PP | 1.088 ± 0.009 | 1.095 ± 0.045 | 0.989 ± 0.006 | 1.119 ± 0.006 | 1.162 ± 0.017 | 1.028 ± 0.032 |
| OTT PETG | 1.091 ± 0.007 | 1.073 ± 0.030 | 0.979 ± 0.018 | 1.147 ± 0.052 | 1.163 ± 0.011 | 1.000 ± 0.012 |
| RID HDPE | 0.936 ± 0.016 | 0.891 ± 0.026 | 0.866 ± 0.011 | 0.754 ± 0.011 | 0.858 ± 0.005 | 0.777 ± 0.010 |
| RID FEP | 0.918 ± 0.058 | 0.882 ± 0.018 | 0.869 ± 0.008 | 0.719 ± 0.005 | 0.843 ± 0.012 | 0.748 ± 0.004 |
| RID PP | 0.886 ± 0.010 | 0.871 ± 0.023 | 0.874 ± 0.009 | 0.738 ± 0.012 | 0.843 ± 0.006 | 0.742 ± 0.005 |
| RID PETG | 0.908 ± 0.019 | 0.855 ± 0.008 | 0.903 ± 0.023 | 0.812 ± 0.106 | 0.850 ± 0.010 | 0.735 ± 0.004 |
| GAT HDPE | 0.866 ± 0.018 | 0.831 ± 0.013 | 0.885 ± 0.026 | 0.853 ± 0.019 | 0.873 ± 0.032 | 0.855 ± 0.006 |
| GAT FEP | 0.853 ± 0.014 | 0.820 ± 0.010 | 0.858 ± 0.004 | 0.821 ± 0.010 | 0.861 ± 0.008 | 0.829 ± 0.027 |
| GAT PP | 0.837 ± 0.006 | 0.831 ± 0.010 | 0.844 ± 0.024 | 0.810 ± 0.013 | 0.881 ± 0.002 | 0.876 ± 0.013 |
| GAT PETG | 0.912 ± 0.080 | 0.833 ± 0.015 | 0.841 ± 0.007 | 0.820 ± 0.002 | 0.885 ± 0.010 | 0.863 ± 0.014 |
| SYN HDPE | 9.670 ± 0.080 | 9.479 ± 0.150 | 9.548 ± 0.043 | 10.079 ± 0.178 | 9.777 ± 0.137 | 9.925 ± 0.027 |
| SYN FEP | 9.558 ± 0.085 | 9.734 ± 0.143 | 9.577 ± 0.133 | 9.971 ± 0.074 | 9.854 ± 0.097 | 9.962 ± 0.043 |
| SYN PP | 9.645 ± 0.048 | 9.626 ± 0.149 | 9.644 ± 0.046 | 10.126 ± 0.034 | 9.807 ± 0.063 | 9.958 ± 0.093 |
| SYN PETG | 9.620 ± 0.070 | 9.398 ± 0.218 | 9.627 ± 0.084 | 10.205 ± 0.077 | 9.824 ± 0.034 | 9.955 ± 0.063 |
| | **Zn** | | | | | |
| SLRS-3 | 1.199 ± 0.023 | 1.178 ± 0.029 | 1.160 ± 0.074 | 1.261 ± 0.026 | 1.200 ± 0.016 | 1.201 ± 0.005 |
| OTT HDPE | 1.822 ± 0.028 | 1.768 ± 0.007 | 1.666 ± 0.033 | 1.895 ± 0.023 | 1.963 ± 0.042 | 1.836 ± 0.014 |
| OTT FEP | 1.921 ± 0.052 | 1.829 ± 0.042 | 1.665 ± 0.020 | 1.867 ± 0.018 | 1.952 ± 0.023 | 1.797 ± 0.017 |
| OTT PP | 1.862 ± 0.012 | 1.870 ± 0.069 | 1.673 ± 0.008 | 1.900 ± 0.020 | 1.974 ± 0.017 | 1.789 ± 0.044 |
| OTT PETG | 1.888 ± 0.033 | 1.840 ± 0.051 | 1.661 ± 0.015 | 1.995 ± 0.142 | 1.960 ± 0.011 | 1.741 ± 0.023 |
| RID HDPE | 1.063 ± 0.013 | 1.029 ± 0.023 | 1.045 ± 0.009 | 0.883 ± 0.023 | 1.043 ± 0.007 | 0.953 ± 0.013 |
| RID FEP | 1.037 ± 0.046 | 1.014 ± 0.009 | 1.034 ± 0.006 | 0.836 ± 0.007 | 1.007 ± 0.011 | 0.918 ± 0.013 |
| RID PP | 1.025 ± 0.023 | 1.022 ± 0.031 | 1.036 ± 0.028 | 0.866 ± 0.022 | 1.029 ± 0.024 | 0.913 ± 0.011 |
| RID PETG | 1.084 ± 0.043 | 0.997 ± 0.019 | 1.087 ± 0.045 | 0.891 ± 0.044 | 1.048 ± 0.026 | 0.929 ± 0.016 |
| GAT HDPE | 2.340 ± 0.006 | 2.272 ± 0.020 | 2.389 ± 0.038 | 2.346 ± 0.009 | 2.343 ± 0.049 | 2.380 ± 0.011 |
| GAT FEP | 2.331 ± 0.012 | 2.258 ± 0.017 | 2.328 ± 0.012 | 2.297 ± 0.005 | 2.322 ± 0.012 | 2.311 ± 0.037 |
| GAT PP | 2.302 ± 0.008 | 2.294 ± 0.011 | 2.316 ± 0.059 | 2.266 ± 0.019 | 2.392 ± 0.025 | 2.471 ± 0.036 |
| GAT PETG | 2.538 ± 0.157 | 2.334 ± 0.041 | 2.308 ± 0.036 | 2.265 ± 0.027 | 2.406 ± 0.034 | 2.431 ± 0.025 |
| SYN HDPE | 10.006 ± 0.086 | 9.856 ± 0.091 | 9.823 ± 0.023 | 10.532 ± 0.138 | 9.761 ± 0.068 | 10.338 ± 0.012 |
| SYN FEP | 9.894 ± 0.084 | 10.091 ± 0.098 | 9.879 ± 0.091 | 10.439 ± 0.050 | 9.819 ± 0.022 | 10.398 ± 0.048 |
| SYN PP | 9.992 ± 0.059 | 9.985 ± 0.150 | 9.912 ± 0.041 | 10.518 ± 0.061 | 9.790 ± 0.174 | 10.381 ± 0.090 |
| SYN PETG | 10.539 ± 0.551 | 9.855 ± 0.324 | 9.937 ± 0.105 | 10.668 ± 0.138 | 9.950 ± 0.118 | 10.670 ± 0.408 |
| | **As** | | | | | |
| SLRS-3 | 0.812 ± 0.005 | 0.779 ± 0.010 | 0.769 ± 0.016 | 0.847 ± 0.021 | 0.790 ± 0.014 | 0.819 ± 0.009 |
| OTT HDPE | 0.488 ± 0.009 | 0.471 ± 0.018 | 0.452 ± 0.003 | 0.509 ± 0.013 | 0.521 ± 0.012 | 0.498 ± 0.005 |
| OTT FEP | 0.500 ± 0.006 | 0.481 ± 0.014 | 0.454 ± 0.002 | 0.499 ± 0.003 | 0.514 ± 0.014 | 0.499 ± 0.010 |
| OTT PP | 0.496 ± 0.005 | 0.490 ± 0.010 | 0.463 ± 0.001 | 0.512 ± 0.005 | 0.526 ± 0.007 | 0.501 ± 0.006 |
| OTT PETG | 0.493 ± 0.001 | 0.487 ± 0.009 | 0.451 ± 0.013 | 0.508 ± 0.009 | 0.515 ± 0.015 | 0.480 ± 0.003 |
| RID HDPE | 0.473 ± 0.006 | 0.447 ± 0.004 | 0.452 ± 0.009 | 0.413 ± 0.012 | 0.466 ± 0.009 | 0.434 ± 0.008 |
| RID FEP | 0.455 ± 0.011 | 0.452 ± 0.008 | 0.454 ± 0.003 | 0.388 ± 0.002 | 0.466 ± 0.011 | 0.428 ± 0.003 |
| RID PP | 0.457 ± 0.010 | 0.444 ± 0.009 | 0.459 ± 0.006 | 0.396 ± 0.003 | 0.481 ± 0.015 | 0.418 ± 0.007 |
| RID PETG | 0.452 ± 0.003 | 0.431 ± 0.010 | 0.458 ± 0.008 | 0.401 ± 0.002 | 0.471 ± 0.017 | 0.417 ± 0.009 |
| GAT HDPE | 0.180 ± 0.001 | 0.168 ± 0.001 | 0.172 ± 0.003 | 0.180 ± 0.002 | 0.182 ± 0.008 | 0.188 ± 0.006 |
| GAT FEP | 0.176 ± 0.003 | 0.168 ± 0.001 | 0.165 ± 0.007 | 0.178 ± 0.004 | 0.179 ± 0.010 | 0.180 ± 0.004 |
| GAT PP | 0.171 ± 0.004 | 0.170 ± 0.007 | 0.170 ± 0.009 | 0.169 ± 0.008 | 0.184 ± 0.006 | 0.191 ± 0.003 |
| GAT PETG | 0.175 ± 0.006 | 0.168 ± 0.007 | 0.167 ± 0.004 | 0.172 ± 0.002 | 0.180 ± 0.004 | 0.181 ± 0.002 |
| SYN HDPE | 9.952 ± 0.055 | 9.637 ± 0.094 | 9.888 ± 0.018 | 10.214 ± 0.091 | 9.229 ± 0.066 | 10.017 ± 0.029 |
| SYN FEP | 9.916 ± 0.031 | 9.848 ± 0.075 | 9.890 ± 0.036 | 10.204 ± 0.029 | 9.456 ± 0.126 | 10.137 ± 0.036 |
| SYN PP | 9.962 ± 0.060 | 9.747 ± 0.138 | 9.879 ± 0.051 | 10.341 ± 0.038 | 9.365 ± 0.110 | 10.132 ± 0.089 |
| SYN PETG | 9.936 ± 0.109 | 9.646 ± 0.193 | 9.852 ± 0.022 | 10.446 ± 0.021 | 9.481 ± 0.021 | 10.192 ± 0.055 |

## Stability study - Appendix 5.1

| | Day 0 | Day 1 | Day 7 | Day 14 | Day 21 | Day 28 |
|---|---|---|---|---|---|---|
| | **Sb** | | | | | |
| SLRS-3 | 0.170 ± 0.003 | 0.162 ± 0.006 | 0.155 ± 0.002 | 0.174 ± 0.003 | 0.161 ± 0.003 | 0.170 ± 0.004 |
| OTT HDPE | 0.260 ± 0.003 | 0.248 ± 0.006 | 0.224 ± 0.002 | 0.258 ± 0.004 | 0.266 ± 0.008 | 0.251 ± 0.004 |
| OTT FEP | 0.270 ± 0.003 | 0.255 ± 0.004 | 0.233 ± 0.002 | 0.254 ± 0.002 | 0.267 ± 0.001 | 0.253 ± 0.001 |
| OTT PP | 0.263 ± 0.005 | 0.258 ± 0.005 | 0.227 ± 0.007 | 0.262 ± 0.003 | 0.267 ± 0.002 | 0.242 ± 0.002 |
| OTT PETG | 0.264 ± 0.002 | 0.264 ± 0.008 | 0.227 ± 0.003 | 0.263 ± 0.004 | 0.267 ± 0.004 | 0.239 ± 0.001 |
| RID HDPE | 0.282 ± 0.004 | 0.271 ± 0.004 | 0.273 ± 0.006 | 0.249 ± 0.001 | 0.276 ± 0.001 | 0.264 ± 0.006 |
| RID FEP | 0.277 ± 0.003 | 0.270 ± 0.004 | 0.282 ± 0.003 | 0.240 ± 0.005 | 0.283 ± 0.004 | 0.260 ± 0.007 |
| RID PP | 0.274 ± 0.004 | 0.265 ± 0.001 | 0.276 ± 0.005 | 0.238 ± 0.007 | 0.276 ± 0.004 | 0.260 ± 0.007 |
| RID PETG | 0.275 ± 0.006 | 0.270 ± 0.003 | 0.281 ± 0.003 | 0.249 ± 0.005 | 0.279 ± 0.004 | 0.253 ± 0.006 |
| GAT HDPE | 0.257 ± 0.010 | 0.248 ± 0.003 | 0.250 ± 0.006 | 0.252 ± 0.003 | 0.244 ± 0.007 | 0.244 ± 0.010 |
| GAT FEP | 0.260 ± 0.001 | 0.253 ± 0.003 | 0.258 ± 0.004 | 0.252 ± 0.002 | 0.253 ± 0.003 | 0.250 ± 0.007 |
| GAT PP | 0.260 ± 0.002 | 0.249 ± 0.004 | 0.249 ± 0.004 | 0.244 ± 0.001 | 0.250 ± 0.002 | 0.257 ± 0.004 |
| GAT PETG | 0.257 ± 0.009 | 0.252 ± 0.004 | 0.251 ± 0.004 | 0.246 ± 0.001 | 0.261 ± 0.004 | 0.263 ± 0.000 |
| SYN HDPE | 10.179 ± 0.187 | 9.754 ± 0.042 | 9.927 ± 0.083 | 10.378 ± 0.120 | 9.744 ± 0.138 | 10.320 ± 0.013 |
| SYN FEP | 10.044 ± 0.111 | 9.792 ± 0.025 | 9.904 ± 0.013 | 10.373 ± 0.031 | 9.881 ± 0.043 | 10.464 ± 0.078 |
| SYN PP | 10.050 ± 0.067 | 9.836 ± 0.076 | 9.940 ± 0.050 | 10.485 ± 0.023 | 9.784 ± 0.082 | 10.390 ± 0.074 |
| SYN PETG | 10.053 ± 0.039 | 9.889 ± 0.101 | 9.851 ± 0.013 | 10.594 ± 0.011 | 9.845 ± 0.031 | 10.385 ± 0.053 |
| | **Tl** | | | | | |
| SLRS-3 | 0.008 ± 0.000 | 0.008 ± 0.000 | 0.008 ± 0.000 | 0.008 ± 0.000 | 0.008 ± 0.000 | 0.008 ± 0.000 |
| OTT HDPE | 0.222 ± 0.003 | 0.216 ± 0.001 | 0.203 ± 0.003 | 0.220 ± 0.002 | 0.226 ± 0.004 | 0.213 ± 0.000 |
| OTT FEP | 0.230 ± 0.001 | 0.216 ± 0.005 | 0.202 ± 0.002 | 0.222 ± 0.001 | 0.224 ± 0.002 | 0.212 ± 0.004 |
| OTT PP | 0.228 ± 0.002 | 0.222 ± 0.006 | 0.202 ± 0.002 | 0.224 ± 0.001 | 0.231 ± 0.002 | 0.204 ± 0.005 |
| OTT PETG | 0.229 ± 0.001 | 0.221 ± 0.005 | 0.196 ± 0.003 | 0.226 ± 0.003 | 0.225 ± 0.001 | 0.197 ± 0.001 |
| RID HDPE | 0.222 ± 0.002 | 0.218 ± 0.003 | 0.223 ± 0.002 | 0.202 ± 0.001 | 0.220 ± 0.003 | 0.209 ± 0.004 |
| RID FEP | 0.219 ± 0.002 | 0.215 ± 0.003 | 0.220 ± 0.001 | 0.190 ± 0.003 | 0.220 ± 0.003 | 0.202 ± 0.002 |
| RID PP | 0.220 ± 0.003 | 0.219 ± 0.002 | 0.225 ± 0.002 | 0.195 ± 0.001 | 0.218 ± 0.002 | 0.201 ± 0.002 |
| RID PETG | 0.214 ± 0.003 | 0.213 ± 0.002 | 0.225 ± 0.001 | 0.195 ± 0.002 | 0.214 ± 0.003 | 0.200 ± 0.002 |
| GAT HDPE | 0.230 ± 0.001 | 0.231 ± 0.002 | 0.239 ± 0.003 | 0.230 ± 0.001 | 0.227 ± 0.004 | 0.228 ± 0.003 |
| GAT FEP | 0.232 ± 0.003 | 0.226 ± 0.001 | 0.234 ± 0.003 | 0.225 ± 0.001 | 0.230 ± 0.002 | 0.222 ± 0.004 |
| GAT PP | 0.230 ± 0.002 | 0.225 ± 0.001 | 0.233 ± 0.003 | 0.219 ± 0.002 | 0.229 ± 0.003 | 0.238 ± 0.004 |
| GAT PETG | 0.226 ± 0.004 | 0.226 ± 0.001 | 0.233 ± 0.001 | 0.217 ± 0.001 | 0.229 ± 0.006 | 0.230 ± 0.003 |
| SYN HDPE | 9.874 ± 0.141 | 9.555 ± 0.064 | 9.794 ± 0.085 | 10.144 ± 0.079 | 9.514 ± 0.077 | 9.894 ± 0.008 |
| SYN FEP | 9.746 ± 0.026 | 9.642 ± 0.041 | 9.701 ± 0.067 | 10.096 ± 0.016 | 9.627 ± 0.110 | 10.014 ± 0.058 |
| SYN PP | 9.719 ± 0.097 | 9.687 ± 0.044 | 9.749 ± 0.045 | 10.216 ± 0.072 | 9.543 ± 0.100 | 10.006 ± 0.062 |
| SYN PETG | 9.700 ± 0.048 | 9.630 ± 0.104 | 9.702 ± 0.026 | 10.325 ± 0.049 | 9.565 ± 0.050 | 9.930 ± 0.087 |
| | **Pb** | | | | | |
| SLRS-3 | 0.073 ± 0.002 | 0.070 ± 0.001 | 0.069 ± 0.001 | 0.075 ± 0.001 | 0.072 ± 0.001 | 0.074 ± 0.001 |
| OTT HDPE | 0.108 ± 0.003 | 0.106 ± 0.001 | 0.097 ± 0.000 | 0.109 ± 0.001 | 0.121 ± 0.015 | 0.105 ± 0.002 |
| OTT FEP | 0.114 ± 0.007 | 0.107 ± 0.003 | 0.097 ± 0.000 | 0.105 ± 0.001 | 0.110 ± 0.002 | 0.103 ± 0.001 |
| OTT PP | 0.109 ± 0.001 | 0.110 ± 0.002 | 0.096 ± 0.000 | 0.107 ± 0.000 | 0.113 ± 0.003 | 0.099 ± 0.003 |
| OTT PETG | 0.111 ± 0.002 | 0.107 ± 0.002 | 0.093 ± 0.002 | 0.113 ± 0.007 | 0.110 ± 0.003 | 0.097 ± 0.001 |
| RID HDPE | 0.075 ± 0.002 | 0.073 ± 0.001 | 0.076 ± 0.000 | 0.065 ± 0.000 | 0.077 ± 0.002 | 0.075 ± 0.008 |
| RID FEP | 0.073 ± 0.002 | 0.073 ± 0.002 | 0.076 ± 0.002 | 0.062 ± 0.001 | 0.075 ± 0.001 | 0.067 ± 0.002 |
| RID PP | 0.074 ± 0.001 | 0.075 ± 0.002 | 0.078 ± 0.000 | 0.062 ± 0.002 | 0.075 ± 0.001 | 0.066 ± 0.000 |
| RID PETG | 0.073 ± 0.002 | 0.073 ± 0.001 | 0.081 ± 0.004 | 0.064 ± 0.002 | 0.077 ± 0.003 | 0.069 ± 0.003 |
| GAT HDPE | 0.079 ± 0.002 | 0.079 ± 0.001 | 0.082 ± 0.000 | 0.078 ± 0.001 | 0.079 ± 0.003 | 0.078 ± 0.001 |
| GAT FEP | 0.080 ± 0.000 | 0.079 ± 0.000 | 0.079 ± 0.001 | 0.076 ± 0.001 | 0.080 ± 0.001 | 0.076 ± 0.001 |
| GAT PP | 0.079 ± 0.002 | 0.080 ± 0.001 | 0.079 ± 0.002 | 0.074 ± 0.001 | 0.080 ± 0.001 | 0.083 ± 0.000 |
| GAT PETG | 0.087 ± 0.004 | 0.084 ± 0.008 | 0.080 ± 0.001 | 0.076 ± 0.002 | 0.083 ± 0.001 | 0.082 ± 0.001 |
| SYN HDPE | 9.837 ± 0.099 | 9.534 ± 0.106 | 9.787 ± 0.070 | 10.117 ± 0.110 | 9.445 ± 0.104 | 9.884 ± 0.020 |
| SYN FEP | 9.762 ± 0.065 | 9.643 ± 0.043 | 9.664 ± 0.075 | 10.089 ± 0.014 | 9.521 ± 0.088 | 10.029 ± 0.076 |
| SYN PP | 9.761 ± 0.051 | 9.661 ± 0.041 | 9.699 ± 0.030 | 10.185 ± 0.029 | 9.560 ± 0.159 | 10.033 ± 0.079 |
| SYN PETG | 9.699 ± 0.097 | 9.640 ± 0.136 | 9.679 ± 0.017 | 10.262 ± 0.057 | 9.623 ± 0.029 | 9.971 ± 0.063 |

| | Day 0 | Day 1 | Day 7 | Day 14 | Day 21 | Day 28 |
|---|---|---|---|---|---|---|
| | **Mn** | | | | | |
| SLRS-3 | 4.1 ± 0.0 | 4.0 ± 0.1 | 4.0 ± 0.1 | 4.1 ± 0.1 | 4.1 ± 0.1 | 4.1 ± 0.0 |
| OTT HDPE | 6.4 ± 0.1 | 6.1 ± 0.1 | 5.8 ± 0.0 | 6.4 ± 0.0 | 6.8 ± 0.2 | 6.3 ± 0.1 |
| OTT FEP | 6.6 ± 0.0 | 6.3 ± 0.1 | 5.8 ± 0.0 | 6.4 ± 0.0 | 6.6 ± 0.0 | 6.2 ± 0.0 |
| OTT PP | 6.5 ± 0.1 | 6.5 ± 0.2 | 5.9 ± 0.0 | 6.5 ± 0.0 | 6.8 ± 0.1 | 6.1 ± 0.2 |
| OTT PETG | 6.5 ± 0.1 | 6.3 ± 0.2 | 5.7 ± 0.1 | 6.6 ± 0.0 | 6.7 ± 0.0 | 5.9 ± 0.0 |
| RID HDPE | 26.5 ± 0.0 | 26.0 ± 0.4 | 26.5 ± 0.1 | 22.1 ± 0.5 | 25.9 ± 0.3 | 24.4 ± 0.3 |
| RID FEP | 25.7 ± 0.3 | 25.7 ± 0.4 | 26.5 ± 0.1 | 21.4 ± 0.2 | 25.7 ± 0.3 | 23.8 ± 0.2 |
| RID PP | 26.3 ± 0.1 | 25.1 ± 0.2 | 26.6 ± 0.1 | 22.1 ± 0.2 | 25.7 ± 0.1 | 23.4 ± 0.1 |
| RID PETG | 26.2 ± 0.0 | 24.6 ± 0.3 | 26.4 ± 0.1 | 22.6 ± 0.2 | 26.0 ± 0.3 | 23.1 ± 0.1 |
| GAT HDPE | 13.3 ± 0.1 | 12.5 ± 0.1 | 13.7 ± 0.4 | 13.0 ± 0.1 | 13.1 ± 0.0 | 13.3 ± 0.1 |
| GAT FEP | 13.3 ± 0.0 | 12.6 ± 0.1 | 13.3 ± 0.1 | 12.7 ± 0.1 | 13.2 ± 0.1 | 13.0 ± 0.3 |
| GAT PP | 13.0 ± 0.1 | 12.8 ± 0.0 | 13.3 ± 0.3 | 12.7 ± 0.1 | 13.4 ± 0.1 | 13.8 ± 0.2 |
| GAT PETG | 12.9 ± 0.2 | 12.7 ± 0.0 | 13.1 ± 0.1 | 12.6 ± 0.0 | 13.4 ± 0.1 | 13.5 ± 0.2 |
| SYN HDPE | 9.7 ± 0.1 | 9.3 ± 0.1 | 9.6 ± 0.1 | 9.9 ± 0.2 | 9.5 ± 0.1 | 9.9 ± 0.0 |
| SYN FEP | 9.6 ± 0.0 | 9.5 ± 0.1 | 9.7 ± 0.2 | 9.8 ± 0.0 | 9.5 ± 0.0 | 10.0 ± 0.1 |
| SYN PP | 9.7 ± 0.1 | 9.5 ± 0.1 | 9.7 ± 0.0 | 10.0 ± 0.1 | 9.5 ± 0.1 | 10.0 ± 0.0 |
| SYN PETG | 9.7 ± 0.1 | 9.4 ± 0.2 | 9.6 ± 0.1 | 10.1 ± 0.1 | 9.6 ± 0.1 | 9.9 ± 0.0 |
| | **Co** | | | | | |
| SLRS-3 | 0.037 ± 0.001 | 0.034 ± 0.002 | 0.023 ± 0.001 | 0.034 ± 0.002 | 0.030 ± 0.006 | 0.027 ± 0.001 |
| OTT HDPE | 0.260 ± 0.002 | 0.247 ± 0.002 | 0.224 ± 0.004 | 0.256 ± 0.002 | 0.265 ± 0.006 | 0.241 ± 0.001 |
| OTT FEP | 0.267 ± 0.001 | 0.255 ± 0.006 | 0.223 ± 0.003 | 0.257 ± 0.004 | 0.260 ± 0.002 | 0.242 ± 0.001 |
| OTT PP | 0.262 ± 0.005 | 0.263 ± 0.009 | 0.228 ± 0.003 | 0.263 ± 0.001 | 0.262 ± 0.006 | 0.237 ± 0.006 |
| OTT PETG | 0.272 ± 0.014 | 0.261 ± 0.007 | 0.226 ± 0.007 | 0.275 ± 0.012 | 0.265 ± 0.005 | 0.234 ± 0.008 |
| RID HDPE | 0.342 ± 0.002 | 0.313 ± 0.012 | 0.258 ± 0.002 | 0.245 ± 0.007 | 0.237 ± 0.003 | 0.226 ± 0.001 |
| RID FEP | 0.323 ± 0.009 | 0.310 ± 0.006 | 0.262 ± 0.007 | 0.235 ± 0.002 | 0.237 ± 0.001 | 0.219 ± 0.002 |
| RID PP | 0.322 ± 0.002 | 0.306 ± 0.004 | 0.267 ± 0.002 | 0.239 ± 0.001 | 0.235 ± 0.005 | 0.213 ± 0.001 |
| RID PETG | 0.330 ± 0.004 | 0.300 ± 0.005 | 0.273 ± 0.008 | 0.249 ± 0.006 | 0.243 ± 0.004 | 0.213 ± 0.005 |
| GAT HDPE | 0.291 ± 0.004 | 0.277 ± 0.002 | 0.287 ± 0.005 | 0.283 ± 0.002 | 0.281 ± 0.004 | 0.282 ± 0.003 |
| GAT FEP | 0.291 ± 0.001 | 0.276 ± 0.002 | 0.281 ± 0.001 | 0.275 ± 0.002 | 0.281 ± 0.002 | 0.275 ± 0.007 |
| GAT PP | 0.284 ± 0.003 | 0.281 ± 0.002 | 0.280 ± 0.008 | 0.276 ± 0.001 | 0.287 ± 0.000 | 0.293 ± 0.005 |
| GAT PETG | 0.293 ± 0.008 | 0.284 ± 0.003 | 0.276 ± 0.005 | 0.274 ± 0.001 | 0.289 ± 0.008 | 0.289 ± 0.003 |
| SYN HDPE | 9.736 ± 0.063 | 9.437 ± 0.127 | 9.540 ± 0.047 | 10.096 ± 0.160 | 9.505 ± 0.047 | 9.878 ± 0.032 |
| SYN FEP | 9.613 ± 0.044 | 9.683 ± 0.107 | 9.531 ± 0.165 | 9.970 ± 0.043 | 9.578 ± 0.098 | 9.957 ± 0.048 |
| SYN PP | 9.741 ± 0.004 | 9.565 ± 0.150 | 9.561 ± 0.053 | 10.070 ± 0.050 | 9.580 ± 0.103 | 9.988 ± 0.045 |
| SYN PETG | 9.721 ± 0.064 | 9.339 ± 0.210 | 9.588 ± 0.083 | 10.070 ± 0.025 | 9.627 ± 0.088 | 9.958 ± 0.048 |
| | **Ni** | | | | | |
| SLRS-3 | 0.722 ± 0.014 | 0.863 ± 0.028 | 0.797 ± 0.008 | 0.943 ± 0.014 | 0.869 ± 0.058 | 0.846 ± 0.014 |
| OTT HDPE | 0.672 ± 0.040 | 0.679 ± 0.016 | 0.598 ± 0.007 | 0.755 ± 0.017 | 0.638 ± 0.017 | 0.660 ± 0.031 |
| OTT FEP | 0.759 ± 0.049 | 0.719 ± 0.023 | 0.588 ± 0.010 | 0.750 ± 0.010 | 0.675 ± 0.052 | 0.672 ± 0.009 |
| OTT PP | 0.698 ± 0.029 | 0.734 ± 0.012 | 0.597 ± 0.021 | 0.770 ± 0.008 | 0.679 ± 0.018 | 0.656 ± 0.006 |
| OTT PETG | 0.697 ± 0.010 | 0.698 ± 0.022 | 0.603 ± 0.006 | 0.765 ± 0.016 | 0.635 ± 0.031 | 0.650 ± 0.018 |
| RID HDPE | 0.513 ± 0.006 | 0.499 ± 0.023 | 0.535 ± 0.040 | 0.470 ± 0.017 | 0.501 ± 0.040 | 0.464 ± 0.015 |
| RID FEP | 0.520 ± 0.041 | 0.512 ± 0.023 | 0.579 ± 0.033 | 0.463 ± 0.009 | 0.454 ± 0.034 | 0.475 ± 0.011 |
| RID PP | 0.505 ± 0.008 | 0.499 ± 0.010 | 0.594 ± 0.029 | 0.458 ± 0.004 | 0.442 ± 0.043 | 0.455 ± 0.007 |
| RID PETG | 0.545 ± 0.017 | 0.492 ± 0.004 | 0.615 ± 0.029 | 0.480 ± 0.022 | 0.441 ± 0.019 | 0.459 ± 0.014 |
| GAT HDPE | 0.519 ± 0.013 | 0.480 ± 0.014 | 0.504 ± 0.020 | 0.557 ± 0.013 | 0.385 ± 0.026 | 0.498 ± 0.013 |
| GAT FEP | 0.525 ± 0.021 | 0.482 ± 0.014 | 0.494 ± 0.010 | 0.541 ± 0.012 | 0.420 ± 0.029 | 0.519 ± 0.037 |
| GAT PP | 0.540 ± 0.024 | 0.482 ± 0.024 | 0.482 ± 0.010 | 0.547 ± 0.012 | 0.412 ± 0.006 | 0.516 ± 0.004 |
| GAT PETG | 0.534 ± 0.037 | 0.504 ± 0.017 | 0.491 ± 0.015 | 0.522 ± 0.023 | 0.421 ± 0.070 | 0.497 ± 0.010 |
| SYN HDPE | 9.783 ± 0.086 | 9.473 ± 0.131 | 9.505 ± 0.054 | 10.103 ± 0.202 | 9.563 ± 0.063 | 9.929 ± 0.060 |
| SYN FEP | 9.674 ± 0.043 | 9.758 ± 0.117 | 9.551 ± 0.178 | 9.950 ± 0.038 | 9.563 ± 0.175 | 9.992 ± 0.094 |
| SYN PP | 9.760 ± 0.022 | 9.648 ± 0.099 | 9.486 ± 0.047 | 10.106 ± 0.021 | 9.411 ± 0.072 | 9.976 ± 0.119 |
| SYN PETG | 9.753 ± 0.042 | 9.490 ± 0.179 | 9.519 ± 0.106 | 10.162 ± 0.088 | 9.492 ± 0.027 | 9.995 ± 0.015 |

| | Day 0 | Day 1 | Day 7 | Day 14 | Day 21 | Day 28 |
|---|---|---|---|---|---|---|
| | **Mo** | | | | | |
| SLRS-3 | 0.232 ± 0.005 | 0.218 ± 0.004 | 0.219 ± 0.003 | 0.238 ± 0.007 | 0.225 ± 0.005 | 0.224 ± 0.011 |
| OTT HDPE | 0.185 ± 0.005 | 0.175 ± 0.002 | 0.165 ± 0.001 | 0.178 ± 0.003 | 0.188 ± 0.007 | 0.167 ± 0.003 |
| OTT FEP | 0.190 ± 0.001 | 0.179 ± 0.003 | 0.165 ± 0.001 | 0.177 ± 0.006 | 0.182 ± 0.003 | 0.163 ± 0.000 |
| OTT PP | 0.185 ± 0.002 | 0.185 ± 0.008 | 0.164 ± 0.001 | 0.183 ± 0.006 | 0.189 ± 0.005 | 0.156 ± 0.002 |
| OTT PETG | 0.185 ± 0.001 | 0.181 ± 0.008 | 0.163 ± 0.004 | 0.181 ± 0.001 | 0.186 ± 0.002 | 0.156 ± 0.001 |
| RID HDPE | 0.554 ± 0.006 | 0.539 ± 0.003 | 0.550 ± 0.002 | 0.482 ± 0.009 | 0.539 ± 0.002 | 0.484 ± 0.007 |
| RID FEP | 0.543 ± 0.001 | 0.530 ± 0.001 | 0.549 ± 0.001 | 0.464 ± 0.005 | 0.529 ± 0.006 | 0.471 ± 0.003 |
| RID PP | 0.531 ± 0.004 | 0.532 ± 0.007 | 0.554 ± 0.004 | 0.479 ± 0.005 | 0.533 ± 0.005 | 0.470 ± 0.008 |
| RID PETG | 0.528 ± 0.002 | 0.515 ± 0.006 | 0.554 ± 0.004 | 0.475 ± 0.005 | 0.533 ± 0.010 | 0.461 ± 0.005 |
| GAT HDPE | 0.093 ± 0.004 | 0.091 ± 0.005 | 0.092 ± 0.004 | 0.086 ± 0.002 | 0.091 ± 0.001 | 0.080 ± 0.005 |
| GAT FEP | 0.087 ± 0.000 | 0.087 ± 0.001 | 0.090 ± 0.002 | 0.082 ± 0.001 | 0.087 ± 0.003 | 0.084 ± 0.003 |
| GAT PP | 0.090 ± 0.002 | 0.090 ± 0.006 | 0.090 ± 0.002 | 0.083 ± 0.002 | 0.091 ± 0.002 | 0.080 ± 0.004 |
| GAT PETG | 0.091 ± 0.004 | 0.085 ± 0.001 | 0.088 ± 0.001 | 0.081 ± 0.002 | 0.088 ± 0.003 | 0.086 ± 0.002 |
| SYN HDPE | 9.712 ± 0.083 | 9.360 ± 0.072 | 9.637 ± 0.056 | 9.915 ± 0.093 | 9.392 ± 0.086 | 9.359 ± 0.058 |
| SYN FEP | 9.615 ± 0.051 | 9.528 ± 0.031 | 9.579 ± 0.038 | 9.911 ± 0.049 | 9.452 ± 0.044 | 9.529 ± 0.029 |
| SYN PP | 9.650 ± 0.022 | 9.517 ± 0.107 | 9.621 ± 0.048 | 10.011 ± 0.052 | 9.438 ± 0.057 | 9.481 ± 0.038 |
| SYN PETG | 9.647 ± 0.048 | 9.408 ± 0.117 | 9.604 ± 0.005 | 10.074 ± 0.023 | 9.457 ± 0.058 | 9.544 ± 0.078 |
| | **Ag** | | | | | |
| SLRS-3 | 0.001 ± 0.001 | 0.002 ± 0.001 | 0.000 ± 0.000 | 0.001 ± 0.000 | 0.003 ± 0.001 | 0.001 ± 0.001 |
| OTT HDPE | 0.206 ± 0.002 | 0.176 ± 0.007 | 0.123 ± 0.010 | 0.128 ± 0.004 | 0.138 ± 0.018 | 0.124 ± 0.017 |
| OTT FEP | 0.213 ± 0.001 | 0.181 ± 0.003 | 0.129 ± 0.011 | 0.100 ± 0.002 | 0.133 ± 0.024 | 0.133 ± 0.008 |
| OTT PP | 0.210 ± 0.004 | 0.191 ± 0.003 | 0.128 ± 0.004 | 0.102 ± 0.002 | 0.147 ± 0.013 | 0.123 ± 0.006 |
| OTT PETG | 0.207 ± 0.002 | 0.186 ± 0.007 | 0.132 ± 0.002 | 0.134 ± 0.000 | 0.152 ± 0.002 | 0.123 ± 0.010 |
| RID HDPE | 0.180 ± 0.002 | 0.174 ± 0.001 | 0.159 ± 0.001 | 0.126 ± 0.006 | 0.157 ± 0.004 | 0.141 ± 0.003 |
| RID FEP | 0.180 ± 0.004 | 0.170 ± 0.001 | 0.159 ± 0.005 | 0.115 ± 0.008 | 0.156 ± 0.004 | 0.144 ± 0.007 |
| RID PP | 0.177 ± 0.005 | 0.170 ± 0.003 | 0.162 ± 0.002 | 0.121 ± 0.001 | 0.154 ± 0.002 | 0.139 ± 0.001 |
| RID PETG | 0.171 ± 0.004 | 0.168 ± 0.004 | 0.165 ± 0.004 | 0.132 ± 0.004 | 0.161 ± 0.005 | 0.138 ± 0.002 |
| GAT HDPE | 0.216 ± 0.001 | 0.188 ± 0.001 | 0.173 ± 0.003 | 0.132 ± 0.000 | 0.136 ± 0.015 | 0.136 ± 0.005 |
| GAT FEP | 0.214 ± 0.004 | 0.191 ± 0.004 | 0.176 ± 0.003 | 0.141 ± 0.004 | 0.150 ± 0.002 | 0.142 ± 0.004 |
| GAT PP | 0.212 ± 0.001 | 0.191 ± 0.002 | 0.177 ± 0.008 | 0.134 ± 0.003 | 0.151 ± 0.006 | 0.152 ± 0.006 |
| GAT PETG | 0.203 ± 0.006 | 0.193 ± 0.002 | 0.181 ± 0.002 | 0.139 ± 0.005 | 0.147 ± 0.002 | 0.142 ± 0.001 |
| SYN HDPE | 9.775 ± 0.063 | 9.409 ± 0.043 | 9.756 ± 0.076 | 10.062 ± 0.089 | 9.549 ± 0.068 | 9.791 ± 0.034 |
| SYN FEP | 9.738 ± 0.056 | 9.552 ± 0.034 | 9.679 ± 0.041 | 9.964 ± 0.024 | 9.603 ± 0.016 | 9.941 ± 0.010 |
| SYN PP | 9.667 ± 0.009 | 9.613 ± 0.117 | 9.748 ± 0.023 | 10.043 ± 0.058 | 9.624 ± 0.089 | 9.920 ± 0.008 |
| SYN PETG | 9.656 ± 0.033 | 9.529 ± 0.096 | 9.599 ± 0.076 | 9.951 ± 0.046 | 9.482 ± 0.135 | 9.680 ± 0.214 |
| | **Cd** | | | | | |
| SLRS-3 | 0.014 ± 0.001 | 0.014 ± 0.001 | 0.014 ± 0.001 | 0.014 ± 0.001 | 0.014 ± 0.000 | 0.013 ± 0.001 |
| OTT HDPE | 0.230 ± 0.004 | 0.225 ± 0.003 | 0.213 ± 0.003 | 0.231 ± 0.002 | 0.241 ± 0.005 | 0.223 ± 0.005 |
| OTT FEP | 0.239 ± 0.001 | 0.228 ± 0.007 | 0.213 ± 0.001 | 0.233 ± 0.002 | 0.240 ± 0.007 | 0.225 ± 0.001 |
| OTT PP | 0.234 ± 0.003 | 0.236 ± 0.009 | 0.213 ± 0.005 | 0.236 ± 0.002 | 0.248 ± 0.002 | 0.220 ± 0.004 |
| OTT PETG | 0.233 ± 0.005 | 0.235 ± 0.008 | 0.209 ± 0.004 | 0.241 ± 0.006 | 0.240 ± 0.004 | 0.212 ± 0.003 |
| RID HDPE | 0.217 ± 0.001 | 0.218 ± 0.003 | 0.223 ± 0.008 | 0.195 ± 0.006 | 0.226 ± 0.003 | 0.204 ± 0.003 |
| RID FEP | 0.215 ± 0.004 | 0.213 ± 0.002 | 0.223 ± 0.003 | 0.184 ± 0.003 | 0.222 ± 0.001 | 0.199 ± 0.003 |
| RID PP | 0.215 ± 0.002 | 0.213 ± 0.005 | 0.224 ± 0.004 | 0.189 ± 0.003 | 0.220 ± 0.003 | 0.198 ± 0.003 |
| RID PETG | 0.209 ± 0.001 | 0.212 ± 0.004 | 0.226 ± 0.004 | 0.191 ± 0.002 | 0.223 ± 0.004 | 0.195 ± 0.001 |
| GAT HDPE | 0.252 ± 0.002 | 0.247 ± 0.000 | 0.261 ± 0.004 | 0.247 ± 0.002 | 0.252 ± 0.002 | 0.249 ± 0.001 |
| GAT FEP | 0.250 ± 0.007 | 0.250 ± 0.006 | 0.253 ± 0.003 | 0.243 ± 0.003 | 0.254 ± 0.001 | 0.246 ± 0.005 |
| GAT PP | 0.248 ± 0.001 | 0.246 ± 0.002 | 0.252 ± 0.004 | 0.240 ± 0.003 | 0.257 ± 0.003 | 0.261 ± 0.007 |
| GAT PETG | 0.245 ± 0.002 | 0.245 ± 0.002 | 0.248 ± 0.001 | 0.236 ± 0.005 | 0.257 ± 0.006 | 0.252 ± 0.002 |
| SYN HDPE | 9.951 ± 0.065 | 9.584 ± 0.061 | 9.880 ± 0.030 | 10.213 ± 0.071 | 9.790 ± 0.102 | 10.130 ± 0.030 |
| SYN FEP | 9.919 ± 0.062 | 9.687 ± 0.049 | 9.772 ± 0.063 | 10.149 ± 0.029 | 9.864 ± 0.038 | 10.211 ± 0.070 |
| SYN PP | 9.879 ± 0.040 | 9.774 ± 0.086 | 9.852 ± 0.029 | 10.197 ± 0.047 | 9.801 ± 0.073 | 10.130 ± 0.032 |
| SYN PETG | 9.889 ± 0.028 | 9.758 ± 0.092 | 9.840 ± 0.011 | 10.307 ± 0.041 | 9.828 ± 0.030 | 10.122 ± 0.039 |

| | Day 0 | Day 1 | Day 7 | Day 14 | Day 21 | Day 28 |
|---|---|---|---|---|---|---|
| | **Hg-0.5% BrCl** | | | | | |
| SLRS-3 | | | | | | |
| OTT HDPE | 0.054 ± 0.001 | 0.054 ± 0.001 | 0.057 ± 0.002 | 0.056 ± 0.001 | 0.056 ± 0.001 | 0.055 ± 0.000 |
| OTT FEP | 0.054 ± 0.001 | 0.054 ± 0.001 | 0.054 ± 0.001 | 0.056 ± 0.001 | 0.055 ± 0.000 | 0.054 ± 0.001 |
| OTT PP | | | | | | |
| OTT PETG | 0.054 ± 0.001 | 0.054 ± 0.001 | 0.055 ± 0.000 | 0.056± 0.001 | 0.057 ± 0.002 | 0.055 ± 0.000 |
| RID HDPE | 0.054 ± 0.001 | 0.053 ± 0.001 | 0.055 ± 0.000 | 0.055 ± 0.000 | 0.055 ± 0.000 | 0.056 ± 0.0002 |
| RID FEP | 0.054 ± 0.001 | 0.051 ± 0.002 | 0.055 ± 0.000 | 0.055 ± 0.000 | 0.055 ± 0.000 | 0.056 ± 0.001 |
| RID PP | | | | | | |
| RID PETG | 0.054 ± 0.001 | 0.054 ± 0.001 | 0.055 ± 0.001 | 0.055 ± 0.001 | 0.055 ± 0.000 | 0.056 ± 0.001 |
| GAT HDPE | 0.054 ± 0.001 | 0.052 ± 0.001 | 0.053 ± 0.000 | 0.055 ± 0.000 | 0.055 ± 0.000 | 0.056 ± 0.000 |
| GAT FEP | 0.054 ± 0.001 | 0.052 ± 0.001 | 0.054 ± 0.000 | 0.054 ± 0.002 | 0.055 ± 0.000 | 0.054 ± 0.001 |
| GAT PP | | | | | | |
| GAT PETG | 0.054 ± 0.001 | 0.052 ± 0.001 | 0.055 ± 0.001 | 0.056± 0.001 | 0.055 ± 0.001 | 0.055 ± 0.001 |
| SYN HDPE | 0.054 ± 0.001 | 0.053 ± 0.000 | 0.053 ± 0.002 | 0.055± 0.001 | 0.054 ± 0.001 | 0.054 ± 0.000 |
| SYN FEP | 0.054 ± 0.001 | 0.053 ± 0.001 | 0.055 ± 0.001 | 0.055± 0.001 | 0.054 ± 0.001 | 0.056 ± 0.001 |
| SYN PP | | | | | | |
| SYN PETG | 0.054 ± 0.001 | 0.054 ± 0.000 | 0.057 ± 0.001 | 0.055± 0.000 | 0.055 ± 0.001 | 0.055 ± 0.000 |
| | **Hg-2% HCl** | | | | | |
| SLRS-3 | | | | | | |
| OTT HDPE | 0.056 ± 0.000 | 0.048 ± 0.001 | 0.047 ± 0.001 | 0.047 ± 0.001 | 0.045 ± 0.001 | 0.043 ± 0.002 |
| OTT FEP | 0.056 ± 0.001 | 0.047 ± 0.002 | 0.049 ± 0.001 | 0.048 ± 0.001 | 0.046 ± 0.000 | 0.044 ± 0.001 |
| OTT PP | | | | | | |
| OTT PETG | 0.056 ± 0.001 | 0.049 ± 0.001 | 0.047 ± 0.001 | 0.046± 0.001 | 0.045 ± 0.002 | 0.043 ± 0.001 |
| RID HDPE | 0.055 ± 0.001 | 0.047 ± 0.001 | 0.048 ± 0.000 | 0.042 ± 0.001 | 0.040 ± 0.001 | 0.043 ± 0.001 |
| RID FEP | 0.055 ± 0.001 | 0.049 ± 0.002 | 0.047 ± 0.001 | 0.042 ± 0.001 | 0.041 ± 0.002 | 0.043 ± 0.002 |
| RID PP | | | | | | |
| RID PETG | 0.055 ± 0.001 | 0.048 ± 0.001 | 0.046 ± 0.001 | 0.042 ± 0.001 | 0.040 ± 0.001 | 0.041 ± 0.002 |
| GAT HDPE | 0.056 ± 0.001 | 0.050 ± 0.001 | 0.051 ± 0.000 | 0.050 ± 0.001 | 0.048 ± 0.001 | 0.046± 0.000 |
| GAT FEP | 0.056 ± 0.001 | 0.050 ± 0.001 | 0.051 ± 0.001 | 0.050 ± 0.001 | 0.050 ± 0.000 | 0.046± 0.001 |
| GAT PP | | | | | | |
| GAT PETG | 0.056 ± 0.001 | 0.050 ± 0.000 | 0.051 ± 0.001 | 0.050 ± 0.001 | 0.049 ± 0.001 | 0.046± 0.001 |
| SYN HDPE | 0.053± 0.001 | 0.051 ± 0.001 | 0.052 ± 0.001 | 0.053± 0.000 | 0.051 ± 0.001 | 0.050 ± 0.001 |
| SYN FEP | 0.053± 0.000 | 0.052 ± 0.001 | 0.052 ± 0.001 | 0.054± 0.001 | 0.050 ± 0.001 | 0.053 ± 0.002 |
| SYN PP | | | | | | |
| SYN PETG | 0.053± 0.001 | 0.052 ± 0.001 | 0.052 ± 0.000 | 0.052± 0.001 | 0.050 ± 0.001 | 0.047 ± 0.002 |
| | **Hg-K2Cr2O7** | | | | | |
| SLRS-3 | | | | | | |
| OTT HDPE | 0.051 ± 0.001 | 0.045 ± 0.002 | 0.040 ± 0.001 | 0.041 ± 0.001 | 0.042 ± 0.001 | 0.042 ± 0.002 |
| OTT FEP | 0.051 ± 0.001 | 0.047 ± 0.002 | 0.043 ± 0.001 | 0.042 ± 0.001 | 0.044 ± 0.001 | 0.044 ± 0.001 |
| OTT PP | | | | | | |
| OTT PETG | 0.051 ± 0.002 | 0.046 ± 0.001 | 0.040 ± 0.001 | 0.042± 0.000 | 0.043 ± 0.002 | 0.044 ± 0.001 |
| RID HDPE | 0.052 ± 0.001 | 0.047 ± 0.001 | 0.047 ± 0.000 | 0.046 ± 0.001 | 0.050 ± 0.001 | 0.051 ± 0.002 |
| RID FEP | 0.052 ± 0.001 | 0.047 ± 0.001 | 0.046 ± 0.001 | 0.046 ± 0.001 | 0.050 ± 0.002 | 0.051 ± 0.002 |
| RID PP | | | | | | |
| RID PETG | 0.052 ± 0.001 | 0.047 ± 0.002 | 0.046 ± 0.001 | 0.043 ± 0.002 | 0.047 ± 0.001 | 0.047 ± 0.002 |
| GAT HDPE | 0.051 ± 0.000 | 0.046 ± 0.001 | 0.045 ± 0.001 | 0.045 ± 0.001 | 0.047 ± 0.001 | 0.048± 0.000 |
| GAT FEP | 0.051 ± 0.001 | 0.046 ± 0.001 | 0.045 ± 0.001 | 0.045 ± 0.000 | 0.047 ± 0.002 | 0.047± 0.002 |
| GAT PP | | | | | | |
| GAT PETG | 0.051 ± 0.001 | 0.046 ± 0.001 | 0.045 ± 0.001 | 0.045 ± 0.001 | 0.045 ± 0.001 | 0.046± 0.001 |
| SYN HDPE | 0.051 ± 0.001 | 0.049 ± 0.001 | 0.051 ± 0.000 | 0.050 ± 0.001 | 0.052 ± 0.001 | 0.051 ± 0.001 |
| SYN FEP | 0.051 ± 0.001 | 0.050 ± 0.001 | 0.051 ± 0.001 | 0.050 ± 0.001 | 0.054 ± 0.002 | 0.052 ± 0.001 |
| SYN PP | | | | | | |
| SYN PETG | 0.051 ± 0.000 | 0.050 ± 0.001 | 0.050 ± 0.001 | 0.049± 0.001 | 0.052 ± 0.001 | 0.052 ± 0.001 |

| | Day 0 | Day 1 | Day 7 | Day 14 | Day 21 | Day 28 |
|---|---|---|---|---|---|---|
| | **Se** | | | | | |
| SLRS-3 | | | | | | |
| OTT HDPE | 0.051 ± 0.002 | 0.058 ± 0.001 | 0.070 ± 0.000 | 0.069 ± 0.002 | 0.063 ± 0.001 | 0.061 ± 0.002 |
| OTT FEP | 0.051 ± 0.002 | 0.062 ± 0.001 | 0.070 ± 0.000 | 0.066 ± 0.000 | 0.062 ± 0.002 | 0.061 ± 0.002 |
| OTT PP | 0.051 ± 0.002 | 0.063 ± 0.003 | 0.070 ± 0.000 | 0.066 ± 0.001 | 0.063 ± 0.003 | 0.062 ± 0.001 |
| OTT PETG | 0.051 ± 0.002 | 0.062 ± 0.002 | 0.071 ± 0.001 | 0.068 ± 0.002 | 0.066 ± 0.003 | 0.064 ± 0.001 |
| RID HDPE | 0.077 ± 0.003 | 0.083 ± 0.001 | 0.095 ± 0.001 | 0.091 ± 0.002 | 0.085 ± 0.001 | 0.085 ± 0.002 |
| RID FEP | 0.077 ± 0.003 | 0.083 ± 0.002 | 0.096 ± 0.001 | 0.088 ± 0.001 | 0.087 ± 0.001 | 0.085 ± 0.001 |
| RID PP | 0.077 ± 0.003 | 0.085 ± 0.003 | 0.095 ± 0.001 | 0.087 ± 0.003 | 0.088 ± 0.001 | 0.085 ± 0.002 |
| RID PETG | 0.077 ± 0.003 | 0.085 ± 0.002 | 0.096 ± 0.001 | 0.090 ± 0.001 | 0.090 ± 0.001 | 0.087 ± 0.000 |
| GAT HDPE | 0.041 ± 0.001 | 0.048 ± 0.001 | 0.053 ± 0.000 | 0.051 ± 0.001 | 0.050 ± 0.001 | 0.045 ± 0.001 |
| GAT FEP | 0.041 ± 0.001 | 0.047 ± 0.001 | 0.054 ± 0.000 | 0.050 ± 0.000 | 0.049 ± 0.002 | 0.045 ± 0.001 |
| GAT PP | 0.041 ± 0.001 | 0.048 ± 0.002 | 0.053 ± 0.000 | 0.051 ± 0.001 | 0.049 ± 0.002 | 0.044 ± 0.001 |
| GAT PETG | 0.041 ± 0.001 | 0.049 ± 0.001 | 0.054 ± 0.002 | 0.049 ± 0.001 | 0.053 ± 0.001 | 0.045 ± 0.001 |
| SYN HDPE | 0.053 ± 0.000 | 0.055 ± 0.002 | 0.057 ± 0.001 | 0.054 ± 0.000 | 0.057 ± 0.001 | 0.052 ± 0.000 |
| SYN FEP | 0.053 ± 0.000 | 0.060 ± 0.001 | 0.056 ± 0.000 | 0.057 ± 0.003 | 0.058 ± 0.003 | 0.051 ± 0.001 |
| SYN PP | 0.053 ± 0.000 | 0.059 ± 0.001 | 0.056 ± 0.000 | 0.057 ± 0.001 | 0.059 ± 0.001 | 0.050 ± 0.001 |
| SYN PETG | 0.053 ± 0.000 | 0.059 ± 0.000 | 0.057 ± 0.001 | 0.056 ± 0.001 | 0.059 ± 0.001 | 0.050 ± 0.000 |